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Volume 1 of 3

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Office of Air Quality Planning and Standards  
Research Triangle Park, NC 27711

LTV Steel Company Indiana Harbor Works East Chicago, Illinois

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# Integrated Iron and Steel Industry

## Final Report Volume I of III

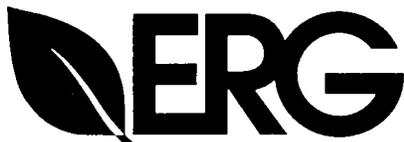
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Indiana Harbor Works  
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E A S T E R N   R E S E A R C H   G R O U P ,   I N C .

## TABLE OF CONTENTS

	<b>Page</b>
1.0 INTRODUCTION .....	1-1
1.1 Objective .....	1-1
1.2 Brief Site Description .....	1-2
1.3 Emissions Measurements Program .....	1-2
1.3.1 Test Matrix .....	1-3
1.3.2 Test Schedule .....	1-3
1.3.3 Sampling Locations .....	1-3
1.3.4 Sampling and Analysis Methods .....	1-6
1.4 Quality Assurance/Quality Control (QA/QC) .....	1-6
1.5 Test Report .....	1-7
2.0 SUMMARY OF RESULTS .....	2-1
2.1 Emissions Test Log .....	2-1
2.2 D/F/PAH Results .....	2-1
2.2.1 Overview .....	2-1
2.2.2 D/F Emission Results .....	2-4
2.2.3 PAH Emission Results .....	2-7
2.3 Metals HAPs Results .....	2-11
2.3.1 Overview .....	2-11
2.3.2 Metal HAPs Emission Results .....	2-11
2.4 PM Results .....	2-16
2.4.1 PM Emissions Results .....	2-16
3.0 PROCESS DESCRIPTION AND PROCESS DATA (Prepared by RTI) .....	3-1
4.0 SAMPLING LOCATIONS .....	4-1
5.0 SAMPLING AND ANALYTICAL PROCEDURES BY ANALYTE .....	5-1
5.1 Particulate Matter and Metals Emissions Testing Using EPA Method 29 ....	5-1
5.1.1 Method 29 Sampling Equipment .....	5-1
5.1.2 Method 29 Sampling Equipment Preparation .....	5-3
5.1.2.1 Glassware Preparation .....	5-3
5.1.2.2 Reagent Preparation .....	5-4
5.1.2.3 Equipment Preparation .....	5-5
5.1.3 Method 29 Sampling Operations .....	5-7
5.1.3.1 Preliminary Measurements .....	5-7
5.1.3.2 Assembling the Train .....	5-7
5.1.3.3 Sampling Procedures .....	5-8

## TABLE OF CONTENTS (Continued)

	Page
5.1.4 Method 29 Sample Recovery .....	5-12
5.1.5 Particulate Analysis .....	5-18
5.1.6 Metals Analytical Procedures .....	5-18
5.1.7 Quality Control for Metals Analytical Procedures .....	5-22
5.1.7.1 ICAP Standards and Quality Control Samples .....	5-23
5.1.7.2 Cold Vapor Atomic Absorption Standards and Quality Control Samples .....	5-23
5.2 CDD/CDF and PAH Emissions Testing Using EPA Method 23 .....	5-24
5.2.1 Method 23 Sampling Equipment .....	5-24
5.2.2 Method 23 Equipment Preparation .....	5-24
5.2.2.1 Glassware Preparation .....	5-26
5.2.2.2 XAD-2® Resin and Filters Preparation .....	5-26
5.2.2.3 Method 23 Sampling Train Preparation .....	5-28
5.2.3 Method 23 Sampling Operations .....	5-29
5.2.3.1 Preliminary Measurements .....	5-29
5.2.3.2 Assembling the Train .....	5-29
5.2.3.3 Sampling Procedures .....	5-30
5.2.4 CDD/CDF/PAH Sample Recovery .....	5-35
5.2.5 CDD/CDF/PAH Analytical Procedures .....	5-38
5.2.5.1 Preparation of Samples for Extraction .....	5-42
5.2.5.2 Calibration of GC/MS System .....	5-42
5.2.6 CDD/CDF Analytical Quality Control .....	5-42
5.2.6.1 CDD/CDF Quality Control Blanks .....	5-43
5.2.6.2 Quality Control Standards and Duplicates .....	5-44
5.2.7 Analytes and Detection Limits for Method 23 .....	5-45
5.3 Analysis of Method 23 Samples for PAHs .....	5-47
5.4 EPA Methods 1-4 .....	5-50
5.4.1 Traverse Point Location By EPA Method 1 .....	5-50
5.4.2 Volumetric Flow Rate Determination by EPA Method 2 .....	5-50
5.4.2.1 Sampling and Equipment Preparation .....	5-50
5.4.2.2 Sampling Operations .....	5-50
5.4.3 O <sub>2</sub> and CO <sub>2</sub> Concentrations by EPA Method 3 .....	5-51
5.4.4 Average Moisture Determination by EPA Method 4 .....	5-51
6.0 QUALITY ASSURANCE/QUALITY CONTROL .....	6-1
6.1 Sampling QC Results .....	6-1
6.1.1 D/F/PAH Sampling QC .....	6-1
6.1.2 Metals/PM Sampling QC .....	6-4

**TABLE OF CONTENTS (Continued)**

	<b>Page</b>
6.2 Analytical QC Results .....	6-11
6.2.1 D/F/PAH Analytical Quality Control .....	6-11
6.2.2 Metals Analytical Quality Control .....	6-12
6.2.3 PM Analytical Quality Assurance .....	6-14

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## LIST OF TABLES

	<b>Page</b>
1-1 Test Matrix, LTV Steel Plant, East Chicago, Illinois .....	1-4
2-1 Emissions Test Log, LTV Steel .....	2-2
2-2 Sample Volume Collected, dscm .....	2-3
2-3 Flue Gas Volumetric Flow Rates, dscmm .....	2-3
2-4 Dioxin/Furan Stack Gas Concentrations, Venturi Outlet .....	2-5
2-5 Dioxin/Furan Stack Emission Rate, Venturi Outlet .....	2-6
2-6 Dioxin/Furan 2,3,7,8-TCDD Toxicity Equivalent Stack Gas Concentrations, Venturi Outlet .....	2-8
2-7 PAH Concentration, Venturi Outlet .....	2-9
2-8 PAH Stack Emission Rate, Venturi Outlet .....	2-10
2-9 Metals Results: Venturi Outlet, Run 1 ( $\mu\text{g}$ collected) .....	2-12
2-10 Metals Results: Venturi Outlet, Run 2 ( $\mu\text{g}$ collected) .....	2-12
2-11 Metals Results: Venturi Outlet, Run 3 ( $\mu\text{g}$ collected) .....	2-13
2-12 Metals Results: Venturi Inlet, Run 1 ( $\mu\text{g}$ collected) .....	2-13
2-13 Metals Results: Venturi Inlet, Run 2 ( $\mu\text{g}$ collected) .....	2-14
2-14 Metals Results: Venturi Inlet, Run 3 ( $\mu\text{g}$ collected) .....	2-14
2-15 Metals Stack Gas Concentration, Venturi Outlet .....	2-15
2-16 Metals Stack Gas Concentration, Venturi Inlet .....	2-15
2-17 Metals Stack Emission Rate, Venturi Outlet .....	2-17
2-18 Venturi Removal Efficiency for Metals .....	2-17

## LIST OF TABLES (Continued)

	<b>Page</b>
2-19	Particulate Matter Concentration, Venturi Scrubber ..... 2-18
2-20	Particulate Matter Emission Rate and Venturi Scrubber Removal Efficiency ..... 2-18
5-1	Glassware Cleaning Procedure (Train Components) ..... 5-4
5-2	Sampling Checklist ..... 5-9
5-3	Analytical Detection Limits ..... 5-17
5-4	Method 29 Detection Limits ..... 5-19
5-5	Method 23 Glassware Cleaning Procedure (Train Components, Sample Containers and Laboratory Glassware) ..... 5-27
5-6	CDD/CDF Sampling Checklist ..... 5-32
5-7	Method 23 Sample Fractions Shipped To Analytical Laboratory ..... 5-38
5-8	CDD/CDF Congeners To Be Analyzed ..... 5-39
5-9	PAH to be Analyzed ..... 5-40
5-10	Method 23 Blanks Collected ..... 5-43
5-11	Analytical Detection Limits For Dioxins/Furans ..... 5-46
5-12	CDD/CDF Method Detection Limits ..... 5-47
5-13	Analytical Detection Limits For PAHs ..... 5-48
5-14	PAH Method Detection Limits ..... 5-49
6-1	Summary of Leak Checks Performed, Per Port, Dioxin Testing, Outlet ..... 6-2
6-2	Summary of Isokinetic Percentages ..... 6-3
6-3	Dry Gas Meter Post Calibration Results ..... 6-5

**LIST OF TABLES (Continued)**

	<b>Page</b>
6-4 Dioxin/Furan Field Blank Analysis Results .....	6-6
6-5 Summary of Leak Checks Performed, Per Port, Metals Testing, Outlet .....	6-7
6-6 Summary of Leak Checks Performed, Per Port, Metals Testing, Inlet .....	6-8
6-7 Metals QC Results: ( $\mu\text{g}$ detected) .....	6-10

## LIST OF FIGURES

	<b>Page</b>
1-1 Test Schedule .....	1-5
4-1 Venturi Inlet Sampling Location .....	4-2
4-2 Venturi Outlet Sampling Location .....	4-3
4-3 Outlet Traverse Point Layout .....	4-4
4-4 Inlet Traverse Point Layout .....	4-5
5-1 EPA Method 29 Sampling Train .....	5-2
5-2 Method 29 Sample Recovery Scheme .....	5-13
5-3 Method 29 Sample Preparation and Analysis Scheme .....	5-20
5-4 Method 23 Sampling Train Configuration .....	5-25
5-5 Method 23 Field Recovery Scheme .....	5-36
5-6 Extraction and Analysis Schematic for Method 23 Samples .....	5-41



## 1.0 INTRODUCTION

Integrated iron and steel manufacturing is among the categories of major sources for which national emission standards for hazardous air pollutants (NESHAPS) are to be issued by November 2000 pursuant to Section 112 of the Clean Air Act. The integrated iron and steel manufacturing category includes mills that produce steel from iron ore. Key processes and unit operations include sinter production, iron production, steel making, continuous casting, and the preparation of semi-finished and finished products.

Source tests are required to quantify and characterize the particulate matter (PM), hazardous air pollutant (HAP) emissions, and the performance of a sintering plant equipped with a venturi scrubber.

### 1.1 Objective

The objective of the testing at the LTV Steel plant in East Chicago, Illinois, was to perform all activities necessary to characterize the venturi-scrubbed sintering plant windbox for the following emission components:

- Particulate mass (PM) and metal HAPs using EPA Method 29; and
- Dioxins/furans (D/F) and polynuclear aromatic hydrocarbons (PAH) using EPA Method 23.

In addition, the determination of total hydrocarbons using Method 25A and preliminary screening for organic HAPs using a Fourier Transform Infrared (FTIR) monitoring instrument were conducted by Midwest Research Institute (MRI) under a separate work assignment. Testing by ERG and MRI occurred simultaneously. The FTIR element is not included within this final report.

Testing was performed at the inlet and outlet simultaneously. ERG coordinated all field test activities with MRI personnel.

## **1.2 Brief Site Description**

The sintering process is used to agglomerate fine raw materials into a product suitable for charging into a blast furnace. Raw materials processed include ore, fines, limestone, coke, flue dust, basic oxygen furnace (BOF) slag, pellet chips, filter cake and mill scale. The principal emission point at a sinter plant is the exhaust from the sintering machine windbox. Emission controls include baghouses and wet venturi scrubbers.

Major process units operated by LTV Steel at the East Chicago, Illinois, location include one sintering machine, two blast furnaces, BOFs, a continuous caster, and several finishing mills.

The plant has a rated capacity of 5,280 tons per day (tpd) of sinter. The plant operates 24 hours per day, 310 days per year (shutdown every other Thursday). Feed materials for the sinter plant are stored in ten storage bins. The feed to the sinter machine consists of slag, ore, scale, lime, flue dust, coke breeze, filter cake, dolomite, slag metallic fines, Heckett fines, and kish fines.

## **1.3 Emissions Measurements Program**

This section provides an overview of the emissions measurements program conducted at LTV Steel Company in East Chicago, Illinois. Included in this section are summaries of the test matrix, sampling locations, sampling methods, and laboratory analysis. Additional detail on these topics is provided in the sections that follow.

### **1.3.1 Test Matrix**

The sampling and analytical matrix is presented in Table 1-1. Manual emissions tests were employed; detailed descriptions of these sampling and analytical procedures are provided in Section 5.0.

### **1.3.2 Test Schedule**

The daily test schedule is presented in Figure 1-1. The test required two days of set-up, three test days, and one tear-down day. Each test day was approximately 12 hours in length with a typical working period being between 6:00 am and 8:00 pm.

The test schedule was based on the test duration assumed in Table 1-1. The only major delay in the schedule occurred during set-up when the plant was shut down while the plant maintenance crew removed the port caps at the inlet location.

### **1.3.3 Sampling Locations**

The stack gas sampling was conducted at the inlet and outlet of the venturi scrubber. The inlet location was a rectangular duct with four existing 4" ports positioned on the long vertical side. A new 3" port was installed by the plant down stream of the existing ports to accommodate the FTIR probe. Access to this location required the construction of a scaffolding platform which was provided by the plant.

The outlet location was a circular stack with four 4" existing ports positioned 90 degrees apart. The installation of an additional port for FTIR sampling was not possible. Therefore, close coordination between ERG and MRI personnel was needed to accommodate the FTIR, as well as the Method 23 and the Method 29 probes simultaneously in three of the four ports with the necessary port changes during the manual methods testing.

**Table 1-1. Test Matrix, LTV Steel Plant, East Chicago, Illinois**

Sample Location	Number Of Runs	Sample Type	Reference Method	Sample Duration*	Analysis Method	Laboratory
Venturi Inlet	3	Gas Velocity/ Volume/Moisture	EPA Methods 1-4	4 Hrs	Volumetric/Gravimetric	ERG
Venturi Inlet	3	Total Particulates/Metals (Pb, Cr, Cd, Be, Ni, Co, As, Sb, Mn, Se, Hg)	EPA Method 29	4 Hrs	Gravimetric/Atomic Absorption/ICAP	ERG and Triangle Labs
Venturi Outlet	3	Gas Velocity/ Volume/Moisture	EPA Methods 1-4	4 Hrs	Volumetric/Gravimetric	ERG
Venturi Outlet	3	Total Particulates/ Metals (Pb, Cr, Cd, Be, Ni, As, Sb, Co, Mn, Se, Hg)	EPA Method 29	4 Hrs	Gravimetric Atomic Absorption/ICAP	ERG and Triangle Labs
Venturi Outlet	3	D/F/PAHs	EPA Method 23	4 Hrs	GC/HRMS, GC/MS 8290/8270	Triangle Labs

# June

1997

1997

SUNDAY	MONDAY	TUESDAY	WEDNESDAY	THURSDAY	FRIDAY	SATURDAY
1	2	3	4	5	6	7
8	9	10	11	12	13	14
15	16	17	18	19	20	21
22 Travel	23 Coordination Meeting with plant personnel and equipment set-up	24 Set-up	25 Test Day #1	26 Test Day #2	27 Test Day #3	28 Travel
29	30					

Figure 1-1. Test Schedule

### **1.3.4 Sampling and Analysis Methods**

Total particulate matter emissions along with 11 metal HAPs (Pb, Ni, Cr, Mn, Se, Be, Sb, Co, Cd, As, and Hg) were determined using a single sampling train following the protocol provided in EPA Method 29. Particulate loading on the filter and the front half rinse (nozzle/probe, front half of the filter holder) was determined gravimetrically. Metals analyses were then performed on the residue from this rinse, the filter and the back-half impinger catch using inductively coupled argon plasma spectroscopy (ICAPS) for all metals except Hg. Cold vapor atomic absorption (CVAA) was used for Hg. Flue gas samples for D/F and PAHs were collected using EPA Method 23. Flue gas was extracted isokinetically and any D/F/PAH was collected on the filter, the XAD-2® resin trap and in the impingers. The analysis was performed using high resolution gas chromatography (HRGC) coupled with high resolution mass spectrometry (HRMS) for D/F, and both GC/HRMS and GC/LRMS for the PAHs.

### **1.4 Quality Assurance/Quality Control (QA/QC)**

All flue gas testing procedures followed comprehensive QA/QC procedures as outlined in the Site Specific Test Plan (SSTP) and the Quality Assurance Project Plan (QAPP). A full description of the resulting QA parameters is given in Section 6.

All post-test and port change leak checks met the criteria prescribed in the manual methods procedure. The allowable isokinetic QC range of  $\pm 10\%$  was met for all D/F/PAH and metals/PM sampling runs. All post-test dry gas meter calibration checks were within 5% of the full calibration factor. Field blanks (FB) for the D/F/PAH tests showed virtually no contamination. However, the metals FB for the inlet and outlet locations did show some contamination for Cr, Mn, Ni, Pb, Sb, and Se, due most likely to laboratory contamination. The metals FB is discussed in detail in Sections 6.1.2 and 6.2.2.

All analyses were completed under a strict QA/QC regimen. For the D/F/PAH results, percent recoveries of all isotopically labeled compounds were within the lower and upper limits of recovery as specified in the method. For the metals results, all matrix spike recoveries were within the acceptable range.

The manual flue gas test data reflected very little variation over the three runs. The percent relative standard deviation (%RSD) for each of the D/F congeners ranged from 2.8 to 32. The %RSD for the metals ranged from 2 to 25 and the gravimetric results ranged from 12 to 14. These values indicate that the process was very stable during the test period.

## **1.5 Test Report**

This final report, presenting all data collected and the results of the analyses, has been prepared in six sections and two volumes as described below:

- Section 1 provides an introduction to the testing effort and includes a brief description of the test site, an overview of the emissions measurements program and a brief overview of the QC results;
- Section 2 gives a summary of the test results for the D/F/PAH, metals and PM tests;
- Section 3 provides a description of the process and plant operation during the field test. These data are to be supplied by EPA;
- Section 4 gives a discussion of the sampling locations;
- Section 5 presents detailed descriptions of the sampling and analysis procedures; and
- Section 6 provides details of the quality assurance/quality control procedures used on this program and the QC results.

The appendices containing copies of the actual field data sheets and the results of the laboratory analyses are contained in a separate volume.



## **2.0 SUMMARY OF RESULTS**

This section provides the results of the emissions test program conducted at the LTV Steel Company sintering operation from June 23 to June 27, 1997. Included in this section are results of manual tests conducted for D/F/PAH, metal HAPs and PM.

### **2.1 Emissions Test Log**

Nine tests were conducted over a three day period (3 D/F/PAH and 6 Metals/PM). Table 2-1 presents the emissions test log which shows the test date, location, run number, test type, run times and port change times for each test method.

Table 2-2 shows the volume of stack gas sampled for each run in dry standard cubic meters (dscm) and Table 2-3 shows the stack gas volumetric flow rate during each run in dry standard cubic meters per minute (dscmm). The percent relative standard deviation (%RSD) calculated for the three runs for each test method (shown in Table 2-3) was less than 3%, indicating that the process flow was very constant over the three test days. All related field data sheets are given in Appendix E.

## **2.2 D/F/PAH RESULTS**

### **2.2.1 Overview**

Three 4-hour D/F/PAH emission test runs were completed at LTV Steel during the week of June 23, 1997. Three test runs were completed at the outlet of the venturi scrubber associated with the sintering plant windbox. The sample collection protocol followed EPA Method 23 while the analysis protocol was modified to allow the analysis of the sample extract for PAHs.

**Table 2-1. Emissions Test Log, LTV Steel**

<b>Date</b>	<b>Location</b>	<b>Run Number</b>	<b>Test Type</b>	<b>Run Time</b>
6/25/97	Outlet, Port C	1	D/F/PAH	0931-1031
	Outlet, Port D	1	D/F/PAH	1211-1311
	Outlet, Port A	1	D/F/PAH	1406-1506
	Outlet, Port B	1	D/F/PAH	1601-1701
	Outlet, Port D	1	Metals/PM	0930-1030
	Outlet, Port A	1	Metals/PM	1210-1310
	Outlet, Port B	1	Metals/PM	1405-1505
	Outlet, Port C	1	Metals/PM	1600-1700
	Inlet, Port A	1	Metals/PM	0931-1031
	Inlet, Port B	1	Metals/PM	1210-1310
	Inlet, Port C	1	Metals/PM	1405-1505
	Inlet, Port D	1	Metals/PM	1600-1700
	6/26/97	Outlet, Port B	2	D/F/PAH
Outlet, Port A		2	D/F/PAH	1126-1226
Outlet, Port D		2	D/F/PAH	1306-1406
Outlet, Port C		2	D/F/PAH	1436-1536
Outlet, Port C		2	Metals/PM	0955-1055
Outlet, Port B		2	Metals/PM	1125-1225
Outlet, Port A		2	Metals/PM	1305-1405
Outlet, Port D		2	Metals/PM	1435-1535
Inlet, Port A		2	Metals/PM	0955-1055
Inlet, Port B		2	Metals/PM	1125-1225
Inlet, Port C		2	Metals/PM	1305-1405
Inlet, Port D		2	Metals/PM	1435-1535
6/27/97		Outlet, Port C	3	D/F/PAH
	Outlet, Port D	3	D/F/PAH	1001-1101
	Outlet, Port A	3	D/F/PAH	1126-1226
	Outlet, Port B	3	D/F/PAH	1246-1346
	Outlet, Port D	3	Metals/PM	0840-0940
	Outlet, Port A	3	Metals/PM	1000-1100
	Outlet, Port B	3	Metals/PM	1125-1225
	Outlet, Port C	3	Metals/PM	1245-1345
	Inlet, Port A	3	Metals/PM	0835-0935
	Inlet, Port B	3	Metals/PM	1000-1100
	Inlet, Port C	3	Metals/PM	1121-1221
	Inlet, Port D	3	Metals/PM	1242-1342

**Table 2-2. Sample Volume Collected, dscm\***

<b>Location</b>	<b>Parameter</b>	<b>Run 1</b>	<b>Run 2</b>	<b>Run 3</b>	<b>Average</b>	<b>%RSD</b>
Outlet	D/F/PAH	4.22	4.24	4.29	4.25	0.85
Outlet	Metals/PM	4.35	4.14	4.16	4.22	2.75
Inlet	Metals/PM	1.76	1.81	1.76	1.78	1.54

\*dscm, dry standard cubic meters. Standard conditions are defined as 1 atm and 68°F

**Table 2-3. Flue Gas Volumetric Flow Rates, dscmm\***

<b>Location</b>	<b>Parameter</b>	<b>Run 1</b>	<b>Run 2</b>	<b>Run 3</b>	<b>Average</b>	<b>%RSD</b>
Outlet	D/F/PAH	7432	7497	7689	7539	1.77
Outlet	Metals/PM	7691	7614	7388	7564	2.08
Inlet	Metals/PM	7026	6826	7238	7030	2.93

\*dscmm, dry standard cubic meters per minute. Standard conditions are defined as 1 atm and 68°F

This modification to the sample preparation procedure and subsequent analysis is discussed in Section 5 of this report.

### **2.2.2 D/F Emission Results**

Table 2-4 presents the concentration, in nanograms per dry standard cubic meter (ng/dscm), for the selected D/F congeners by run number, the average concentration over the three runs and the %RSD. All results except for the 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF) were determined by high resolution gas chromatography (HRGC)/high resolution mass spectrometry (HRMS) using a DB-5 capillary gas chromatographic column. The 2,3,7,8-TCDF was determined by HRGC/HRMS using a DB-225 column which gives improved chromatographic resolution for this compound over the DB-5 and thus a more accurate quantitation.

As noted in Table 2-4, the reported concentration of several congeners may be over-estimated due to the presence of an associated diphenyl ether (DPE) that coelutes with the peak of interest. However, these values are at or very near the detection limit for that compound or they are very consistent with the value(s) from the other test runs that do not have this DPE interferent and should be considered as estimated maximum possible concentrations (EMPC). These values are included in all calculations. Any compound that was not detected is reported as a “less than value” with this value being the instrumental detection limit. A “less than” value rather than a “0” is used in all appropriate calculations. The %RSDs reported in Table 2-4 for the three runs by compound are generally less than 15% indicating excellent reproducibility. In a few cases, the %RSDs are higher where the concentrations are near the detection limit or the presence of a DPE is indicated. Increased variability would not be unusual in these cases.

Table 2-5 shows the D/F stack emission rates from the venturi outlet. These values were calculated from the average concentrations from Table 2-4 and the average stack flow rate from Table 2-3.

**Table 2-4. Dioxin/Furan Stack Gas Concentrations, Venturi Outlet**

Congener	ng/dscm				
	Run 1	Run 2	Run 3	Average	%RSD
2,3,7,8 -TCDD	0.019 <sup>3</sup>	0.019 <sup>3</sup>	0.014 <sup>3</sup>	0.017 <sup>3</sup>	16.4
1,2,3,7,8-PeCDD	0.050	0.050	0.047	0.049	3.5
1,2,3,4,7,8-HxCDD	0.009 <sup>3</sup>	0.014 <sup>1,3</sup>	0.009 <sup>3</sup>	0.011 <sup>3</sup>	25.1
1,2,3,6,7,8-HxCDD	0.222	0.210	0.217	0.216	2.8
1,2,3,7,8,9-HxCDD	0.114	0.099	0.093	0.102	10.2
1,2,3,4,6,7,8--HpCDD	0.260	0.260	0.219	0.246	9.5
1,2,3,4,6,7,8,9-OCDD	0.196	0.168	0.163 <sup>1</sup>	0.176	10.2
2,3,7,8-TCDF <sup>2</sup>	0.260	0.210	0.170	0.214	21.1
1,2,3,7,8-PeCDF	0.140	0.127	0.098	0.122	17.6
2,3,4,7,8-PeCDF	0.132	0.132	0.107	0.124	11.6
1,2,3,4,7,8-HxCDF	0.158	0.163	0.126	0.149	13.5
1,2,3,6,7,8-HxCDF	0.059	0.064	0.051	0.058	10.8
2,3,4,6,7,8-HxCDF	0.054	0.057	0.040	0.050	18.4
1,2,3,7,8,9-HxCDF	0.005 <sup>1,3</sup>	0.007 <sup>3</sup>	<0.007	0.006 <sup>3</sup>	21.3
1,2,3,4,6,7,8-HpCDF	0.088	0.090	0.084	0.087	3.3
1,2,3,4,7,8,9-HpCDF	0.012 <sup>3</sup>	0.014 <sup>1,3</sup>	0.012 <sup>1,3</sup>	0.013 <sup>3</sup>	11.1
1,2,3,4,6,7,8,9-OCDF	0.038 <sup>3</sup>	0.024 <sup>1,3</sup>	0.047 <sup>3</sup>	0.036 <sup>3</sup>	32.3

<sup>1</sup> Maximum value, may include interference from a diphenyl ether

<sup>2</sup> Determined from DB-225 GC column

<sup>3</sup> Amount detected is less than 5 times the detection limit and should be considered only an estimate

**Table 2-5. Dioxin/Furan Stack Emission Rate, Venturi Outlet**

Congener	Average Concentration ng/dscm	Average Emission Rate µg/Hr
2,3,7,8 -TCDD	0.017 <sup>3</sup>	7.69 <sup>3</sup>
1,2,3,7,8-PeCDD	0.049	22.2
1,2,3,4,7,8-HxCDD <sup>1</sup>	0.011 <sup>3</sup>	4.98 <sup>3</sup>
1,2,3,6,7,8-HxCDD	0.216	97.7
1,2,3,7,8,9-HxCDD	0.102	46.1
1,2,3,4,6,7,8--HpCDD	0.246	111
1,2,3,4,6,7,8,9-OCDD <sup>1</sup>	0.176	79.6
2,3,7,8-TCDF <sup>2</sup>	0.214	96.8
1,2,3,7,8-PeCDF	0.122	55.2
2,3,4,7,8-PeCDF	0.124	56.1
1,2,3,4,7,8-HxCDF	0.149	67.4
1,2,3,6,7,8-HxCDF	0.058	26.2
2,3,4,6,7,8-HxCDF	0.050	22.6
1,2,3,7,8,9-HxCDF <sup>1</sup>	0.006 <sup>3</sup>	2.71 <sup>3</sup>
1,2,3,4,6,7,8-HpCDF	0.087	39.4
1,2,3,4,7,8,9-HpCDF <sup>1</sup>	0.013 <sup>3</sup>	5.88 <sup>3</sup>
1,2,3,4,6,7,8,9-OCDF <sup>1</sup>	0.036 <sup>3</sup>	16.3 <sup>3</sup>

<sup>1</sup> Maximum value, may include interference from a diphenyl ether

<sup>2</sup> Determined from DB-225 GC column

<sup>3</sup> Amount detected is less than 5 times the detection limit and should be considered only an estimate

Table 2-6 shows the congener concentrations in ng/dscm converted to 2,3,7,8-tetrachlorodibenzo-*p*-dioxin toxicity equivalents as well as a summation of the values presented as total chlorinated dioxins and total chlorinated furans. All D/F analytical raw data can be found in Appendix A.

### **2.2.3 PAH Emission Results**

Table 2-7 presents the concentration, in micrograms per dry standard cubic meter ( $\mu\text{g}/\text{dscm}$ ), for the selected PAH compounds by run number, the average concentration over the three runs and the %RSD. All sample extracts were initially analyzed by high resolution chromatography (HRGC)/high resolution mass spectrometry (HRMS) using a DB-5 capillary gas chromatographic column. However, the high levels of many of the PAHs saturated the HRMS resulting, for the most part, in data that were mostly qualitative in nature. Therefore, the extracts were reanalyzed on a low resolution mass spectrometer (LRMS) after dilution of the sample extracts. The %RSDs reported in Table 2-7 for the three runs by compound are generally less than 15% indicating excellent reproducibility. In a few cases, the %RSDs are higher where the concentrations are near the detection limit. Increased variability would not be unusual in this case.

Table 2-8 shows the PAH stack emission rates from the venturi outlet. These values were calculated from the average concentrations from Table 2-7 and the average stack flow rate from Table 2-3. All PAH analytical raw data can be found in Appendix B.

**Table 2-6. Dioxin/Furan 2,3,7,8-TCDD Toxicity Equivalent Stack Gas Concentrations, Venturi Outlet**

Congener	2,3,7,8-CDD TEF <sup>4</sup>	ng/dscm			
		Run 1	Run 2	Run 3	Average
2,3,7,8 -TCDD	1.0	0.019	0.019	0.014	0.017 <sup>3</sup>
1,2,3,7,8-PeCDD	0.5	0.025	0.025	0.024	0.025
1,2,3,4,7,8-HxCDD	0.1	0.0009	0.0014 <sup>1</sup>	0.0009	0.0011 <sup>3</sup>
1,2,3,6,7,8-HxCDD	0.1	0.0222	0.0210	0.0217	0.0216
1,2,3,7,8,9-HxCDD	0.1	0.0114	0.0099	0.0093	0.0102
1,2,3,4,6,7,8--HpCDD	0.01	0.00260	0.00260	0.00219	0.00246
1,2,3,4,6,7,8,9-OCDD	0.001	0.000196	0.000168	0.000163 <sup>1</sup>	0.000176
<b>Total PCDD</b>					<b>0.0775</b>
2,3,7,8-TCDF <sup>2</sup>	0.1	0.0260	0.0210	0.017	0.0213
1,2,3,7,8-PeCDF	0.05	0.007	0.0064	0.0049	0.0056
2,3,4,7,8-PeCDF	0.5	0.066	0.0066	0.054	0.062
1,2,3,4,7,8-HxCDF	0.1	0.0158	0.0163	0.0126	0.0149
1,2,3,6,7,8-HxCDF	0.1	0.0059	0.0064	0.0051	0.0058
2,3,4,6,7,8-HxCDF	0.1	0.0054	0.0057	0.0040	0.0050
1,2,3,7,8,9-HxCDF	0.1	0.0005 <sup>1</sup>	0.0007	<0.0007	0.0006 <sup>3</sup>
1,2,3,4,6,7,8-HpCDF	0.01	0.00088	0.00090	0.00084	0.00087
1,2,3,4,7,8,9-HpCDF	0.01	0.00012	0.00014 <sup>1</sup>	0.00012 <sup>1</sup>	0.00013 <sup>3</sup>
1,2,3,4,6,7,8,9-OCDF	0.001	0.000038	0.000024 <sup>1</sup>	0.000047	0.000036 <sup>3</sup>
<b>Total PCDF</b>					<b>0.1162</b>

<sup>1</sup> Maximum value, may include interference from a diphenyl ether

<sup>2</sup> Determined from DB-225 GC column

<sup>3</sup> The amount detected is less than 5 times the detection limit and should be considered only an estimate

<sup>4</sup> TEF, Toxicity Equivalent Factor

**Table 2-7. PAH Concentration, Venturi Outlet**

PAHs	Concentration, $\mu\text{g}/\text{dscm}$				
	Runs			Average	%RSD
	1	2	3		
Naphthalene*	76.5	90.0	68.5	78.3	13.9
2-Methylnaphthalene*	29.5	31.7	25.4	28.9	11.1
2-Chloronaphthalene	0.038	0.040	0.040	0.039	2.94
Acenaphthylene	8.35	8.62	5.86	7.61	20.0
Acenaphthene	3.24	3.98	3.23	3.48	12.3
Fluorene	5.31	6.18	4.72	5.40	13.6
Phenanthrene*	44.8	46.2	37.1	42.7	11.5
Anthracene	1.73	1.82	1.83	1.79	3.07
Fluoranthene	7.82	7.40	5.44	6.89	18.4
Pyrene	3.25	3.45	2.42	3.04	18.0
Benzo(a)anthracene	0.545	0.618	0.413	0.525	19.8
Chrysene	1.49	1.49	1.00	1.33	21.6
Benzo(b)fluoranthene	1.32	1.40	0.856	1.19	24.6
Benzo(k)fluoranthene	0.223	0.241	0.196	0.220	10.3
Benzo(e)pyrene	0.846	0.835	0.597	0.759	18.5
Benzo(a)pyrene	0.239	0.274	0.187	0.233	18.8
Perylene	0.043	0.071	0.061	0.058	24.3
Indeno(1,2,3-cd)pyrene	0.256	0.309	0.212	0.259	18.8
Dibenzo(a,h)anthracene	0.107	0.094	0.090	0.097	9.16
Benzo(g,h,i)perylene	0.360	0.413	0.296	0.356	16.4

\*Concentrations taken from a sample dilution; should be considered estimated.

**Table 2-8. PAH Stack Emission Rate, Venturi Outlet**

<b>PAHs</b>	<b>Average Concentration (<math>\mu\text{g}/\text{dscm}</math>)</b>	<b>Emission Rate (g/hr)</b>
Naphthalene*	78.3	35.4
2-Methylnaphthalene*	28.9	13.1
2-Chloronaphthalene	0.039	0.018
Acenaphthylene	7.61	3.44
Acenaphthene	3.48	1.58
Fluorene	5.40	2.44
Phenanthrene*	42.7	19.3
Anthracene	1.79	0.811
Fluoranthene	6.89	3.12
Pyrene	3.04	1.38
Benzo(a)anthracene	0.525	0.238
Chrysene	1.33	0.599
Benzo(b)fluoranthene	1.19	0.539
Benzo(k)fluoranthene	0.220	0.100
Benzo(e)pyrene	0.759	0.343
Benzo(a)pyrene	0.233	0.106
Perylene	0.058	0.026
Ideno(1,2,3-cd)pyrene	0.259	0.117
Dibenzo(a,h)anthracene	0.097	0.044
Benzo(g,h,i)perylene	0.356	0.161

\*Concentrations taken from a sample dilution; should be considered estimated.

## **2.3 Metals HAPs Results**

### **2.3.1 Overview**

Six 4-hour metals emission test runs were completed at LTV Steel during the week of June 23, 1997. Three test runs were completed at the inlet and three at the outlet of the venturi scrubber associated with the sintering plant windbox. The sample collection protocol followed EPA Method 29 using a single sampling train to determine emission rates of 11 metal HAPs. A total of five (5) fractions for each test run were presented to the laboratory for analysis (see Section 5 of this report for details).

### **2.3.2 Metal HAPs Emission Results**

Tables 2-9 through 2-14 show the results of the analysis, by fraction, for each of the three samples collected at the outlet and at the inlet along with a total amount detected. Any metal that was not detected is reported as a "less than" value with this value being the instrument detection limit. A "less than" value rather than a "0" is used in all appropriate calculations. Using the results shown in Tables 2-9 through 2-14 and the sample volume collected in the corresponding train given in Table 2-2, the concentration of each metal was calculated. The concentration ( $\mu\text{g}/\text{dscm}$ ) of each metal by run number, the average concentration and %RSD for the outlet and inlet are given in Tables 2-15 and 2-16, respectively. There is an apparent analysis problem associated with manganese for Run 3 outlet ( $103 \mu\text{g}/\text{dscm}$ ) and with cobalt for Run 1 inlet ( $0.051 \mu\text{g}/\text{dscm}$ ). These two values are not consistent with their other two associated test run results. Sample contamination from field activities can be ruled out due to the obvious consistency between runs for each of the other metals. If these two values are removed from the data set, the average concentration given in Table 2-15 for manganese would be  $17.3 \mu\text{g}/\text{dscm}$  and the average concentration given in Table 2-16 for cobalt would be  $0.422 \mu\text{g}/\text{dscm}$ .

**Table 2-9. Metals Results: Venturi Outlet, Run 1 (µg collected)**

Metal	Fraction #					Total
	1	2	3	4	5	
Hg	<0.400	<2.68	<0.100	1.37	<0.360	4.91
As	3.96	<0.588				4.55
Be	<0.100	<0.118				<0.218
Cd	76.6	0.658				77.3
Co	<0.100	<0.118				<0.218
Cr	24.5	3.27				27.8
Mn	54.2	11.7				65.9
Ni	86.3	4.61				90.9
Pb	15,900	10.9				15911
Sb	7.23	<0.470				7.70
Se	18.7	21.6				40.3

**Table 2-10. Metals Results: Venturi Outlet, Run 2 (µg collected)**

Metal	Fraction #					Total
	1	2	3	4	5	
Hg	<0.400	<2.64	<0.240	1.81	<2.60	7.69
As	4.33	<0.589				4.92
Be	<0.100	<0.118				<0.218
Cd	67.5	<0.118				67.6
Co	<0.100	<0.118				<0.218
Cr	17.2	1.46				18.7
Mn	56.1	24.4				80.5
Ni	117	0.507				118
Pb	15,000	1.09				15,000
Sb	5.81	<0.471				6.28
Se	18.7	15.8				34.5

**Table 2-11. Metals Results: Venturi Outlet, Run 3 (µg collected)**

Metal	Fraction #					Total
	1	2	3	4	5	
Hg	<0.400	<2.76	<0.180	2.94	<0.360	6.64
As	3.90	<0.585				4.49
Be	<0.100	<0.117				<0.217
Cd	72.7	<0.117				72.8
Co	<0.100	<0.117				<0.217
Cr	17.4	1.99				19.4
Mn*	115	314				429
Ni	66.4	0.509				66.9
Pb	16,300	1.95				16,300
Sb	6.30	0.471				6.77
Se	16.8	18.9				35.7

\*Questionable Data

**Table 2-12. Metals Results: Venturi Inlet, Run 1 (µg collected)**

Metal	Fraction #					Total
	1	2	3	4	5	
Hg	<0.400	<2.44	0.551	<0.840	<0.280	4.51
As	14.2	<0.598				14.8
Be	<0.100	<0.120				<0.220
Cd	86.1	0.882				87.0
Co*	<0.100	<0.120				<0.220
Cr	84.2	7.04				91.2
Mn	1,330	52.3				1,382
Ni	119	4.79				124
Pb	19,300	96.7				19,400
Sb	12.5	<0.478				13.0
Se	34.7	15.1				49.8

\*Questionable Data

**Table 2-13. Metals Results: Venturi Inlet, Run 2 (µg collected)**

Metal	Fraction #					Total
	1	2	3	4	5	
Hg	0.469	<1.76	<0.240	<1.02	<0.260	3.75
As	18.4	<0.647				19.0
Be	<0.100	<0.129				<0.229
Cd	80.8	<0.129				80.9
Co	1.38	<0.129				1.50
Cr	101	1.64				103
Mn	1,800	6.65				1,800
Ni	82.9	1.15				84.1
Pb	18,500	1.39				18,500
Sb	9.93	<0.518				10.4
Se	40.6	20.9				61.5

**Table 2-14. Metals Results: Venturi Inlet, Run 3 (µg collected)**

Metal	Fraction #					Total
	1	2	3	4	5	
Hg	0.547	<1.76	<0.200	<1.04	<0.380	3.93
As	20.1	<0.647				20.7
Be	<0.100	<0.129				<0.229
Cd	84.8	<0.129				84.9
Co	1.87	<0.129				2.0
Cr	99.8	1.99				102
Mn	1,900	12.6				1,910
Ni	87.0	0.475				87.5
Pb	19,100	1.34				19,100
Sb	9.06	<0.518				9.58
Se	32.2	20.5				52.7

**Table 2-15. Metals Stack Gas Concentration, Venturi Outlet**

Metal	µg/dscm				
	Run 1	Run 2	Run 3	Average	%RSD
Hg	1.13	1.86	1.60	1.53	24.2
As	1.05	1.19	1.08	1.10	6.74
Be	0.050	0.053	0.052	0.052	2.61
Cd	17.8	16.3	17.5	17.2	4.46
Co	0.050	0.053	0.052	0.050	2.61
Cr	6.37	4.52	4.66	5.18	19.9
Mn	15.2	19.4	103*	45.9	108
Ni	20.9	28.5	16.1	21.8	28.7
Pb	3658	3623	3919	3733	4.33
Sb	1.77	1.52	1.63	1.64	7.75
Se	9.26	8.33	8.58	8.73	5.52

\*Questionable Data

**Table 2-16. Metals Stack Gas Concentration, Venturi Inlet**

Metal	µg/dscm				
	Run 1	Run 2	Run 3	Average	%RSD
Hg	1.04	0.91	0.94	0.96	7.0
As	3.40	4.59	4.98	4.32	19.0
Be	0.051	0.055	0.055	0.054	4.96
Cd	20.0	19.5	20.4	20.0	2.17
Co	0.051*	0.362	0.481	0.30	74.6
Cr	21.0	24.9	24.5	23.5	9.2
Mn	318	437	460	405	19.0
Ni	28.5	20.3	21.0	23.3	19.5
Pb	4459	4469	4592	4507	1.64
Sb	2.99	2.51	2.30	2.60	13.5
Se	11.5	14.9	12.7	13.0	13.3

\*Questionable Data

Using this corrected value for manganese and the other listed values from Table 2-15 and the average stack flow rate from Table 2-3 , the average emission rate from the venturi outlet for each metal can be calculated. These results, in grams per hour, are given in Table 2-17. Using these values from Table 2-17 in conjunction with the equivalent values for the inlet (see Table 2-18), a removal efficiency for the venturi scrubber was calculated for each metal. All metal analytical raw data are given in Appendix C.

## **2.4 PM Results**

### **2.4.1 PM Emissions Results**

Particulate matter emissions were determined from the same sampling trains used for the collection of metals at the inlet and outlet of the venturi scrubber. Before metals analysis, PM collected on the filter and in the front half acetone rinse (nozzle, probe, front-half filter holder) was analyzed gravimetrically. PM stack gas concentrations, in grams per dry standard cubic meter (g/dscm), the average and %RSD for the three test runs at the inlet and outlet are presented in Table 2-19. The %RSD for both the inlet and outlet were less than 15, showing excellent reproducibility for the sampling and analysis method as well as constant process conditions over the 3 day test period.

Table 2-20 shows the average PM emission rate to be 482 pounds per hour (lb/hr). This value was calculated from the average outlet concentration from Table 2-19 and the average stack flow rate from Table 2-3. Using this value in conjunction with the equivalent value for the inlet (see Table 2-20), a PM removal efficiency for the venturi scrubber was calculated to be 92.1%. The PM analytical raw data are given in Appendix D.

**Table 2-17. Metals Stack Emission Rate, Venturi Outlet**

Metal	Average Concentration µg/dscm	Average Emission Rate g/hr
Hg	1.53	0.693
As	1.10	0.501
Be	0.052	0.023
Cd	17.2	7.81
Co	0.050	0.023
Cr	5.18	2.35
Mn*	17.3	7.85
Ni	21.8	9.91
Pb	3733	1,690
Sb	1.64	0.743
Se	8.73	3.96

\*Average of two test runs.

**Table 2-18. Venturi Removal Efficiency for Metals**

Metal	Average Inlet Rate g/hr	Average Outlet Rate g/hr	Removal Efficiency %
Hg	0.406	0.693	-70.8
As	1.82	0.501	72.5
Be	0.023	0.023	-3.6
Cd	8.43	7.81	7.4
Co	0.178*	0.023	87.1
Cr	9.89	2.35	76.2
Mn	171	7.85*	95.4
Ni	9.82	9.91	-0.90
Pb	1901	1694	10.9
Sb	1.10	0.743	32.2
Se	5.48	3.96	27.7

\*Average using data from two test runs.

**Table 2-19. Particulate Matter Concentration, Venturi Scrubber**

<b>Location</b>	<b>Run 1 g/dscm</b>	<b>Run 2 g/dscm</b>	<b>Run 3 g/dscm</b>	<b>Average g/dscm</b>	<b>%RSD</b>
Outlet	0.033	0.038	0.044	0.038	14.5
Inlet	0.451	0.531	0.575	0.519	12.1

**Table 2-20. Particulate Matter Emission Rate and Venturi Scrubber Removal Efficiency**

<b>Parameter</b>	<b>Average Inlet Rate* lb/hr</b>	<b>Average Outlet Rate* lb/hr</b>	<b>Removal Efficiency %</b>
PM	482	38.0	92.1

\*Average of 3 test runs

### **3.0 LTV Steel's Sinter Plant**

#### **3.1 Overview**

The primary purpose of the sinter plant is to recover the iron value from waste materials generated at iron and steel plants by converting the materials to a product that can be used in the blast furnace (as burden material). Many of these wastes have little or no value otherwise and would require disposal if they could not be recycled by this process. A secondary purpose of the sinter plant is to incorporate blast furnace flux into the sinter rather than charging it separately into the furnace. Limestone wastes are converted to lime on the sinter grate, and the lime is used as a fluxing agent in the blast furnace. The raw material feed (sinter mix) consists of iron ore fines, chips from iron ore pellets, fine limestone, slag from the steelmaking furnace, scale from the steel rolling mill, residue from air and water pollution control equipment (blast furnace flue dust and filter cake), coke breeze (undersize coke that cannot be used in the blast furnace), and steel reverts.

There are currently 9 sinter plants in operation in the U.S. A total of 5 of these plants use scrubbers to control emissions from the sinter plant windbox, and 4 use a baghouse. The sinter plant at LTV Steel in East Chicago, IN, was chosen for testing to evaluate hazardous air pollutants and emission control performance associated with sinter plants that use scrubbers.

#### **3.2 Process Description**

LTV Steel's sinter plant at their Indiana Harbor Works was constructed in 1959 and is a part of the integrated iron and steel plant that also includes blast furnaces, basic oxygen furnaces (BOFs), ladle metallurgy, continuous casting, rolling mills, and galvanizing lines. The sinter plant has a maximum rated capacity of 5,280 tons per day (tpd) and operates 24 hours per day, 7 days a week. Typically, the plant produces 3,800 tpd and operates 24 hours per day for about 310 days per year. The sinter

machine is 8 feet wide and 168 feet long. The major processing steps in the sinter plant include preparation of the sinter mix (feed material), sintering, discharge end operations (crushing and screening), and cooling of the sinter product. Figure 3-1 is a simplified schematic of the sintering process.

The typical feed composition of the sinter mix during the emission tests is shown in Table 3-1.

**TABLE 3-1. SUMMARY OF SINTER MIX (FEED) COMPONENTS**

Feed material	Percent of total for the day		
	Test 1 (6/25/97)	Test 2 (6/26/97)	Test 3 (6/27/97)
Pellet chips (ore)	41.1	40.9	41.3
Mill scale	13.2	14.3	14.4
Limestone	16.6	15.9	15.8
Flue dust	2.7	3.0	3.0
Coke breeze	0.8	0.8	0.9
BOF slag	9.1	9.1	8.9
Fines	7.4	8.2	7.6
NMT blend	3.8	3.2	4.2
Filter cake	5.3	4.6	3.9

The raw materials are fed from 10 storage bins by a table feeder onto a moving belt. This raw feed is mixed in a pug mill, where water is added to create the desired consistency in the mix. A "hearth layer" of material, which is undersize sinter material that is recycled from the screening operation, is first deposited on the grate bars of the sinter pallets, and then the feed mix is added to a depth of about 14 inches.

The sinter feed passes through an ignition furnace that is 12 feet long. The furnace has 9 side burners fueled by natural gas that ignite the surface of the sinter

feed. The sinter pallets move continually through the ignition furnace at about 90 to 100 inches per minute over 21 vacuum chambers called "windboxes." A vacuum is

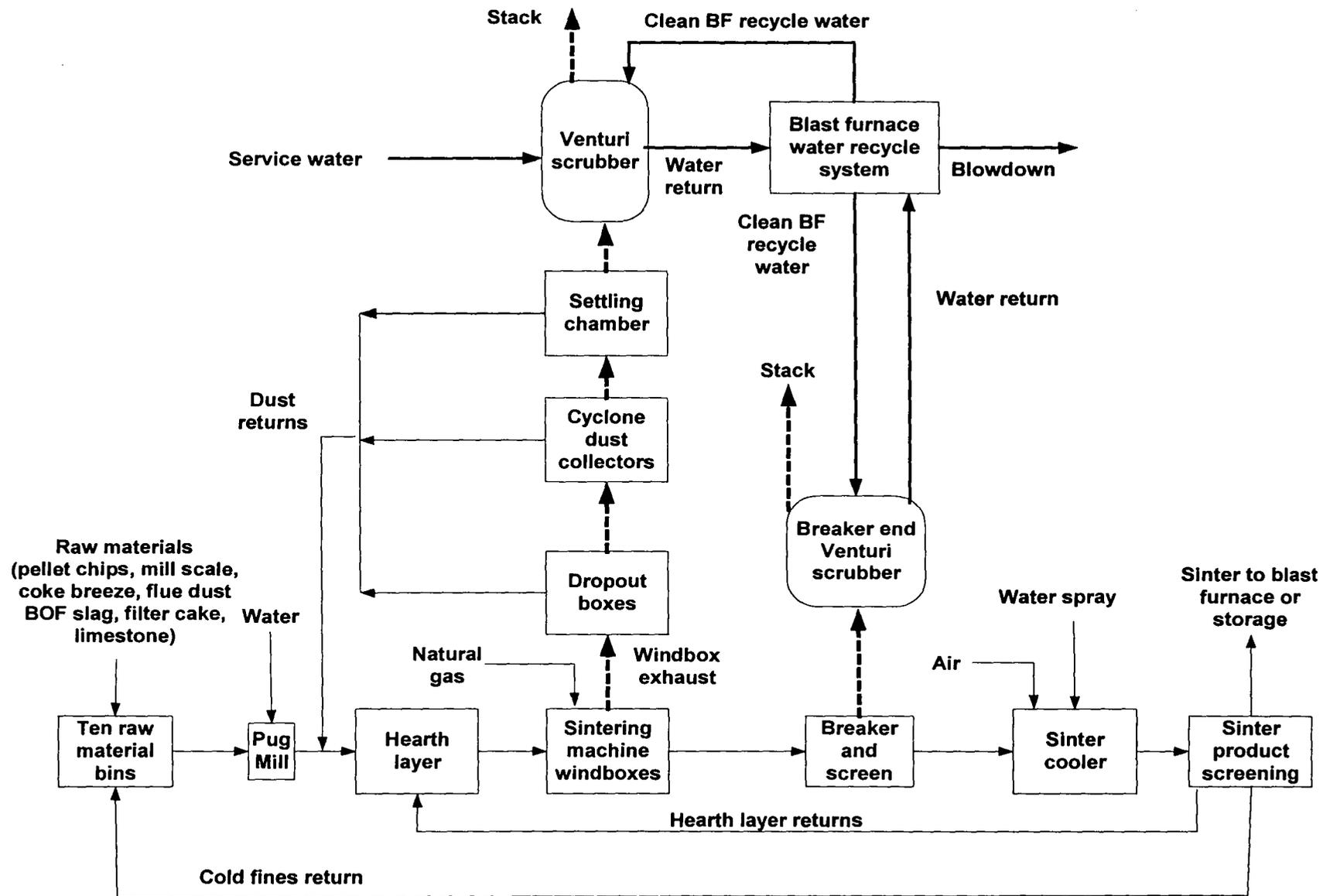


FIGURE 3-1. SCHEMATIC OF MATERIAL FLOW IN THE SINTER PLANT

created in the windbox by a 3,000 horsepower fan that draws heat through the sinter bed and creates the fused "sintered" product.

The red hot sinter from the furnace continues to be transported on the pallets to the breaker, where it is crushed, screened, and discharged to a rotary cooler. Three fans are used to create a forced draft to cool the hot sinter product. In addition, water sprays are used to cool the sinter and to suppress surface dust. The sinter is removed from the bottom of the cooler with a plow that deposits the cooled material onto a conveyor belt. The sinter is then conveyed over a double-deck screen and subsequently deposited into a storage bin. An ore car is used to transport the finished product to the blast furnace. Sinter material that passes through the screens ("fines") is returned to the sinter process for use as the hearth layer or for addition to the sinter mix.

Several operating parameters are monitored and controlled to ensure proper operation of the sinter machine. These parameters include the feed rate of each of the ten feed bins, the sinter furnace temperature, the temperature profile through the various windboxes, draft on the windboxes, speed of the grate, and percent water in the feed. The oil in the sinter feed, which comes primarily from rolling mill scale, is limited to 0.2 percent. During the testing, the coke feed rate appeared to be the parameter that was most often adjusted in order to control temperatures. To maintain the proper chemistry in the blast furnace, an important quality control parameter that is monitored and graphed on a control chart is the percent excess base:

$$(\%CaO + \%MgO) - (\%SiO_2 + \%Al_2O_3).$$

The sinter composition for the 3 tests days is summarized in Table 3-2 and shows that the percent excess base ranged from 13.6 to 13.7 compared to a target of 14.0.

**TABLE 3-2. SUMMARY OF SINTER COMPOSITION**

Component	Percent of total		
	Test 1 (6/25/97)	Test 2 (6/26/97)	Test 3 (6/27/97)
Fe	52.8	52.7	52.9
SiO <sub>2</sub>	4.5	4.6	4.4
Al <sub>2</sub> O <sub>3</sub>	0.59	0.65	0.66
CaO	16.7	16.8	16.6
MgO	2.0	2.1	2.1
Excess base	13.6	13.7	13.6

### 3.4 Emission Control Equipment

Emissions are generated in the process as sinter dust and combustion products and are discharged through the grates and windboxes to a common collector main. Coarse dust particles settle out of the air stream in the collector main and are discharged through flapper valves to a conveyor belt. This conveyor also receives the returns from a series of hoppers that collect any particles that fall under the sinter machine. This material is returned by conveyor to the sinter mix feed for recycle to the process. The exhaust then passes through a battery of cyclones and a series of chambers (originally designed for an electrostatic precipitator that is no longer used). The cyclones and chambers remove dust particles, which are also deposited onto a conveyor (through air actuated valves) for recycle to the process. The exhaust is moved by a 6,000 horsepower fan to the primary control device, which is a double-throat Kinpactor scrubber designed by American Air Filter. The parameters associated with the scrubber that are monitored include the pressure drop across the scrubber, flow rate of water to the scrubber, exhaust fan draft and amperage, and the scrubber water blowdown rate.

Typical operating conditions associated with the scrubber are summarized in Table 3-3.

**TABLE 3-3. TYPICAL SCRUBBER PARAMETERS**

<b>Parameter</b>	<b>Typical value</b>
Liquid/gas ratio	14 gal/1,000 acfm
Water flow rate	3,100 gal/min
Gas flow rate	265,000 scfm
Pressure drop	38 to 46 inches of water
pH of scrubber water	8
Inlet temperature	235 to 270°F
Outlet temperature	120°F
Blowdown rate	240 gal/min

A scrubber is also used to control emissions from the discharge end (i.e., breaker, screens). The discharge end scrubber was not evaluated as part of this test program.

Current State regulations limit particulate matter to 0.02 gr/dscf and 20 percent opacity (6-minute average) for both scrubbers. In addition, the windbox scrubber is limited to a mass rate of 49.7 lb/hr and the discharge end scrubber is limited to 18.05 lb/hr.

### **3.5 Monitoring Results During the Tests**

The operating parameters associated with the process and control device were recorded at 15-minute intervals throughout each test day. The process parameters that were monitored included the feed rate from each of the 10 bins that were used in the

sinter mix, the temperatures and the fan draft for the windboxes, percent water in the feed, sinter machine speed, and the sinter production rate. The emission control device parameters that were monitored included the pressure drop across the scrubber, the water flow rate, blowdown rate, fan draft, and fan amps. Tables 3-4 and 3-5 present a summary of the range of values for these parameters for each test period.

The process and control device appeared to be stable throughout the three test days; consequently, sampling was conducted under normal and representative conditions. The feed rates of mill scale and other materials were typical of the historical rates in recent years that had been reported by the plant. In addition, the oil content of the mill scale was typical (target is 0.2 percent, maximum) with an average of 0.21 percent oil (a range of 0.17 to 0.24 percent) based on the analysis of 5 samples. An examination of the monitoring data showed that the average pressure drop across the scrubber was 43.1, 42.8, and 42.4 inches of water for the 3 test days. The coke rate seemed to be the most variable parameter during the tests because adjustments were made frequently to change the sintering temperature. The coke rate for the 3 tests averaged 1.7, 1.15, and 0.67 tph; consequently, the emission test results may provide some insight into the effect of coke rate on emissions. The windbox temperatures also varied somewhat during the tests. Using Windbox 20 as an example, the average temperatures during the 3 tests were 538, 567, and 443°F.

### **3.6 Analysis of Monitoring and Test Results**

Table 3-6 summarizes the emission results for each run along with selected parameters that were monitored during the test. Only a few comparisons can be made because the process operated stably and consistently during the 3 test runs. One difference is that the coke (fuel) rate during Run 3 was only 39 percent of the rate during Run 1 and only 58 percent of the rate during Run 2. The lower fuel rate during Run 3 is reflected in the lower windbox temperature during Run 3, which was about 100°F lower than in the previous 2 runs. The pollutants most likely to be affected by

the change in combustion conditions are dioxins, furans, and PAHs. During Run 3, the emission rates for all of these compounds were lower than in the previous 2 runs.

The highest emissions of particulate matter and lead occurred during Run 3. The cause is not conclusive, but some of the possible factors affecting this, perhaps in combination, were that Run 3 had the highest sinter feed and production rate and the lowest average pressure drop across the scrubber. In addition, Table 3-4 indicates that Run 3 had a higher feed rate of fines (pellet fines and BOF slag fines) than that recorded during the previous 2 runs. Service water was used in the scrubber during Run 1 and recycled blast furnace water was used during Runs 2 and 3. There is no obvious difference in emissions that can be clearly attributed to the type of scrubber water.

The major metal HAP that was found was lead, which accounted for over 97 percent of the total metal HAP emissions. Discussions with the plant and examination of data from the analysis of blast furnace fines and sludge indicated that a likely source of the lead emissions was from this fine material recycled from the blast furnace. Data in the literature showed that the lead content of blast furnace dust and sludge was generally in the range of 0.01 to 0.1 percent. At a typical feed rate for the dust and sludge of 28,000 lb/hr (14 tph), these materials would introduce 2.8 to 28 lb/hr of lead into the process, which could easily account for the lead that was found entering the scrubber (4.2 lb/hr). In addition, the small particle size of these pollution control residues from the blast furnace may increase the probability that they become airborne, and the volatility of lead and some lead compounds from combustion processes may tend to increase the concentration of lead in the windbox emissions.

Another interesting result is the very low emission rate of dioxins, relative to what had been reported from testing at German sinter plants. For example, the German study reported concentrations of 23 to 68 ng TEQ/m<sup>3</sup> from their initial studies and a range of 5 to 10 ng TEQ/m<sup>3</sup> for plants that optimized and improved their operation. The

results for this sinter plant was much lower with an average concentration of 0.19 ng TEQ/m<sup>3</sup>. On the basis of sinter production, the Germans reported emission levels in the range of 10 to 100 μg/Mg of sinter compared to a measured level of 0.6 μg/Mg of sinter for this plant. The LTV sinter plant had emissions of dioxins and furans that were on the order of 10 to 100 times less than that reported for German sinter plants.

The dioxin results are not unexpected because there are basic differences between the operation of LTV's sinter plant and the German plants. The German study attributed the formation of dioxin to the presence of chlorinated organics, primarily in cutting oils, that were in the waste materials fed to the sintering process. In addition, they stated that the use of electrostatic precipitators contributed to recombination and formation of dioxin. In contrast, the LTV plant has eliminated the purchase and use of chlorinated organics in their facility as part of a voluntary program of pollution prevention, and any new chemical purchases must be approved by the environmental department. Their rolling mill oils (lubricants and hydraulic fluids) do not contain chlorinated compounds. In addition, routine analysis of waste materials going to the sinter plant have not detected chlorinated solvent. Finally, the LTV plant does not use an electrostatic precipitator. Consequently, dioxin rates at LTV that are much lower than those reported by German sinter plants appear to be reasonable and explainable.

Table 3-7 through 3-9 presents a summary of the annual emissions and the emission factors derived from this test.

**TABLE 3-4. PROCESS PARAMETER VALUES DURING THE TESTS**

Parameter	Test 1 (6/25/97)	Test 2 (6/26/97)	Test 3 (6/27/97)
<b>Feed rate (tph):</b>			
Mill scale	25.2 (24.8 - 25.5)	25.2 (24.9 - 25.5)	25.2 (24.8 - 25.6)
BOF slag/filter cake	16.7 (16.1 - 17.9)	16.9 (15.9 - 18.2)	16.9 (15.5 - 17.9)
Fines	16.7 (16.1 - 17.6)	16.4 (15.9 - 18.0)	16.7 (15.3 - 18.0)
Pellet chips	77.4 (75.9 - 78.8)	77.7 (76.2 - 79.0)	77.6 (76.5 - 79.5)
Pellet fines-- blend	9.5 (8.5 - 10.2)	10.7 (10.1 - 11.4)	12.3 (11.3 - 13.6)
Limestone	27.2 (26.9 - 27.7)	27.5 (26.8 - 27.8)	27.7 (27.4 - 28.8)
Cold fines	19.6 (17.6 - 21.4)	17.2 (15.2 - 19.5)	17.8 (16.8 - 23.2)
Coke breeze	1.7 (1.5 - 1.9)	1.2 (0.9 - 1.5)	0.7 (0.34 - 1.1)
Flue dust	5.9 (5.8 - 6.0)	5.9 (5.8 - 6.0)	5.9 (5.8 - 6.0)
BOF slag fines	7.9 (7.6 - 8.2)	9.3 (9.4 - 10.1)	10.0 (9.8 - 10.1)
<b>Other parameters:</b>			
Percent water	6.7 - 7.5	6.5 - 7.4	7.2 - 8.2
Grate speed	70 - 76	70 - 76	70 - 82
Windbox 20 temperature (°F)	453 - 656	474 - 659	334 - 571
Windbox draft (in. water)	13.6 - 17.4	13.3 - 18.2	14.2 - 18.2
Feed rate (tph)	205 - 210	201 - 212	209 - 213
Sinter production (tph)	155 - 158	153 - 161	159 - 161

**TABLE 3-5. CONTROL DEVICE OPERATING PARAMETERS DURING THE TESTS**

Parameter	Test 1 (6/25/97)	Test 2 (6/26/97)	Test 3 (6/27/97)
Pressure drop (in. water)	38.4 - 46.6	39.4 - 46.3	39.8 - 47.0
Water flow (gal/min)	3,040 - 3,085	3,080 - 3,130	3,080 - 3,110
Blowdown (gal/min)	236 - 239	242 - 246	241 - 244
Fan amps	663 - 695	685 - 700	700 - 730
Fan draft (in. water)	3.1 - 5.8	3.2 - 5.8	3.8 - 5.1
Type of water	service (lake)	recycled blast furnace	

**TABLE 3-6. SUMMARY OF RESULTS FOR EACH TEST RUN**

Parameter	Units	Run 1	Run 2	Run 3	Average
PM <sup>a</sup> - inlet	lb/hr	419	479	550	483
PM - outlet	lb/hr	34	38	43	38
PM efficiency	percent	92	92	92	92
Lead - inlet	lb/hr	4.1	4.0	4.4	4.2
Lead - outlet	lb/hr	3.7	3.6	3.8	3.7
Lead efficiency	percent	9.8	10	14	12
HAP metals - in	lb/hr	4.5	4.5	4.9	4.6
HAP metals - out	lb/hr	3.8	3.7	3.9	3.8
Metals efficiency	percent	16	18	20	17
D/F congeners <sup>b</sup>	μg/hr	810	768	<b>694</b>	757
D/F TEQ <sup>c</sup>	μg/hr	93	91	<b>79</b>	88
Total D/F <sup>d</sup>	μg/hr	5,650	5,380	<b>4,820</b>	5,280
7 PAHs <sup>e</sup>	g/hr	1.9	2.0	<b>1.4</b>	1.7
16 PAHs	g/hr	69	78	<b>61</b>	69
TOTAL PAHs	g/hr	83	92	<b>73</b>	83
Sinter feed	tons/hr	208	208	211	209
Sinter production	tons/hr	156	159	160	158
Scrubber Δ p	in. water	43.1	42.8	42.4	42.8
Windbox 20 temperature	°F	538	567	<b>443</b>	516
Coke feed	tons/hr	1.7	1.2	<b>0.7</b>	1.2

<sup>a</sup> PM = particulate matter

<sup>b</sup> D/F congeners are those dioxins and furans that have a toxicity equivalent factor relative to 2,3,7,8-TCDD.

<sup>c</sup> D/F TEQ is the toxicity equivalent expressed relative to 2,3,7,8-TCDD.

<sup>d</sup> Total D/F are all dioxins and furans that were reported.

<sup>e</sup> PAH = polycyclic aromatic hydrocarbons.

**TABLE 3-7 SUMMARY OF RESULTS FOR PM AND HAP METALS**

Pollutant	Concentration (gr/dscf)		Emission rate (lb/hr)		Efficiency (%)	Annual rate (tpy) <sup>a</sup>		Emission factor (lb/t sinter)	
	Inlet	Outlet	Inlet	Outlet		Inlet	Outlet	Inlet	Outlet
Particulate matter	0.23	0.017	483	38	92	1,800	142	3.1	0.24
Pollutant: HAP metals	Concentration (µg/DSCM)		Emission rate (g/hr)		Efficiency (%)	Annual rate (tpy)		Emission factor (lb/t sinter)	
	Inlet	Outlet	Inlet	Outlet		Inlet	Outlet	Inlet	Outlet
Mercury	0.96	1.5	0.41	0.69	0	$3.3 \times 10^{-3}$	$5.7 \times 10^{-3}$	$5.7 \times 10^{-6}$	$9.7 \times 10^{-6}$
Arsenic	4.3	1.1	1.8	0.50	73	$1.5 \times 10^{-2}$	$4.1 \times 10^{-3}$	$2.5 \times 10^{-5}$	$7.0 \times 10^{-6}$
Beryllium	0.054	0.052	0.023	0.023	0	$1.9 \times 10^{-4}$	$1.9 \times 10^{-4}$	$3.2 \times 10^{-7}$	$3.3 \times 10^{-7}$
Cadmium	20	17	8.4	7.8	7.4	$6.9 \times 10^{-2}$	$6.4 \times 10^{-2}$	$1.2 \times 10^{-4}$	$1.1 \times 10^{-4}$
Cobalt	0.30	0.050	0.18	0.023	87	$1.5 \times 10^{-3}$	$1.9 \times 10^{-4}$	$2.5 \times 10^{-6}$	$3.3 \times 10^{-7}$
Chromium	24	5.2	9.9	2.4	76	$8.1 \times 10^{-2}$	$1.9 \times 10^{-2}$	$1.4 \times 10^{-4}$	$3.3 \times 10^{-5}$
Manganese	400	17	171	7.9	95	1.4	$6.4 \times 10^{-2}$	$2.4 \times 10^{-3}$	$1.1 \times 10^{-4}$
Nickel	23	22	9.8	9.9	0	$8.0 \times 10^{-2}$	$8.1 \times 10^{-2}$	$1.4 \times 10^{-4}$	$1.4 \times 10^{-4}$
Lead	4,500	3,700	1,900	1,690	11	16	$1.4 \times 10^{11}$	$2.7 \times 10^{-2}$	$2.4 \times 10^{-2}$
Antimony	2.6	1.6	1.1	0.75	32	$9.0 \times 10^{-3}$	$6.1 \times 10^{-3}$	$1.5 \times 10^{-5}$	$1.0 \times 10^{-5}$
Selenium	13	8.7	5.5	4.0	28	$4.5 \times 10^{-2}$	$3.2 \times 10^{-2}$	$7.7 \times 10^{-5}$	$5.5 \times 10^{-5}$
Total HAP metals	5,000	3,800	2,100	1,700	18	17	$1.4 \times 10^{11}$	$2.9 \times 10^{-2}$	$2.4 \times 10^{-2}$

<sup>a</sup> Based on operation for 24 hours per day for 310 days per year.

**TABLE 3-8 SUMMARY OF RESULTS FOR PAHS**

Pollutant: PAHs <sup>a</sup>	Concentration ( $\mu\text{g}/\text{DSCM}$ )	Emission rate (g/hr)	Annual emissions <sup>b</sup>	
			tpy	lb/ton sinter
Benzo(a)anthracene	0.53	0.24	0.0019	$3.3 \times 10^{-6}$
Chrysene	1.3	0.60	0.0049	$8.4 \times 10^{-6}$
Benzo(b)fluoranthene	1.2	0.54	0.0044	$7.5 \times 10^{-6}$
Benzo(k)fluoranthene	0.22	0.10	0.00082	$1.4 \times 10^{-6}$
Benzo(a)pyrene	0.23	0.11	0.00086	$1.5 \times 10^{-6}$
Indeno(1,2,3-cd)pyrene	0.26	0.12	0.00096	$1.6 \times 10^{-6}$
Dibenzo(a,h)anthracene	0.097	0.044	0.00036	$6.1 \times 10^{-7}$
<b>Total 7 PAHs</b>	<b>3.9</b>	<b>1.7</b>	<b>0.014</b>	<b><math>2.4 \times 10^{-5}</math></b>
Naphthalene	78	35	0.29	$4.9 \times 10^{-4}$
Acenaphthylene	7.6	3.4	0.028	$4.8 \times 10^{-5}$
Acenaphthene	3.5	1.6	0.013	$2.2 \times 10^{-5}$
Fluorene	5.4	2.4	0.020	$3.4 \times 10^{-5}$
Phenanthrene	43	19	0.16	$2.7 \times 10^{-4}$
Anthracene	1.8	0.81	0.0067	$1.1 \times 10^{-5}$
Fluoranthene	6.9	3.1	0.026	$4.3 \times 10^{-5}$
Pyrene	3.0	1.4	0.011	$1.9 \times 10^{-5}$
Benzo(g,h,i)perylene	0.36	0.16	0.0013	$2.2 \times 10^{-6}$
<b>Total 16 PAHs</b>	<b>153</b>	<b>69</b>	<b>0.57</b>	<b><math>9.7 \times 10^{-4}</math></b>
2-Methylnaphthalene	29	13	0.11	$1.8 \times 10^{-4}$
2-Chloronaphthalene	0.039	0.018	0.00015	$2.5 \times 10^{-7}$
Benzo(e)pyrene	0.76	0.30	0.0028	$4.8 \times 10^{-6}$
Perylene	0.058	0.026	0.00022	$3.7 \times 10^{-7}$
<b>Total - all PAHs</b>	<b>183</b>	<b>83</b>	<b>0.68</b>	<b><math>1.2 \times 10^{-3}</math></b>

<sup>a</sup> PAH = polycyclic aromatic hydrocarbons.

<sup>b</sup> Based on operation for 24 hours per day for 310 days per year.

**TABLE 3-9. SUMMARY OF RESULTS FOR DIOXINS AND FURANS**

Pollutant	Concentration (ng/DSCM)	Emission rate ( $\mu$ g/hr)	Annual emissions <sup>a</sup>	
			g/yr	lb/ton sinter
D/F TEQ <sup>b</sup>	0.19	88	0.66	$1.2 \times 10^{-9}$
D/F Congeners <sup>c</sup>	1.7	757	5.6	$1.1 \times 10^{-8}$
D/F Total <sup>d</sup>	11.7	5,280	39	$7.4 \times 10^{-8}$

<sup>a</sup> Based on operation for 24 hours per day for 310 days per year.

<sup>b</sup> D/F TEQ is the toxicity equivalent expressed relative to 2,3,7,8-TCDD.

<sup>c</sup> D/F congeners are those dioxins and furans that have a toxicity equivalent factor relative to 2,3,7,8-TCDD.

<sup>d</sup> Total D/F are all dioxins and furans that were reported.

## 4.0 SAMPLING LOCATIONS

The sampling locations used during the emission testing program at the LTV Steel, East Chicago, Illinois, plant are described in this section. Flue gas samples were collected at the inlet and outlet of the sintering plant wet venturi scrubber using four ports at each location. The configurations of the sampling locations are shown in Figures 4-1 and 4-2.

The test ports and their locations met the requirements of EPA Method 1. The inlet location is a rectangular duct with dimensions of 5' 3" by 10' 10" with four 4" ports installed on the vertical 10' side. The outlet location is a circular stack with an inside diameter (I.D.) of 12 feet with four 4" ports positioned 90 degrees apart. The position and number of traverse points for the outlet and inlet locations are shown in Figures 4-3 and 4-4, respectively. A new sampling port for FTIR sampling was installed at the inlet. Due to the risk of damage to the refractory of the outlet stack, the installation of a new port for the FTIR was not possible. Therefore, the FTIR probe was positioned in one of the existing four ports at the start of a test run and was moved to one of the ports not occupied by the manual methods probes during each port change.

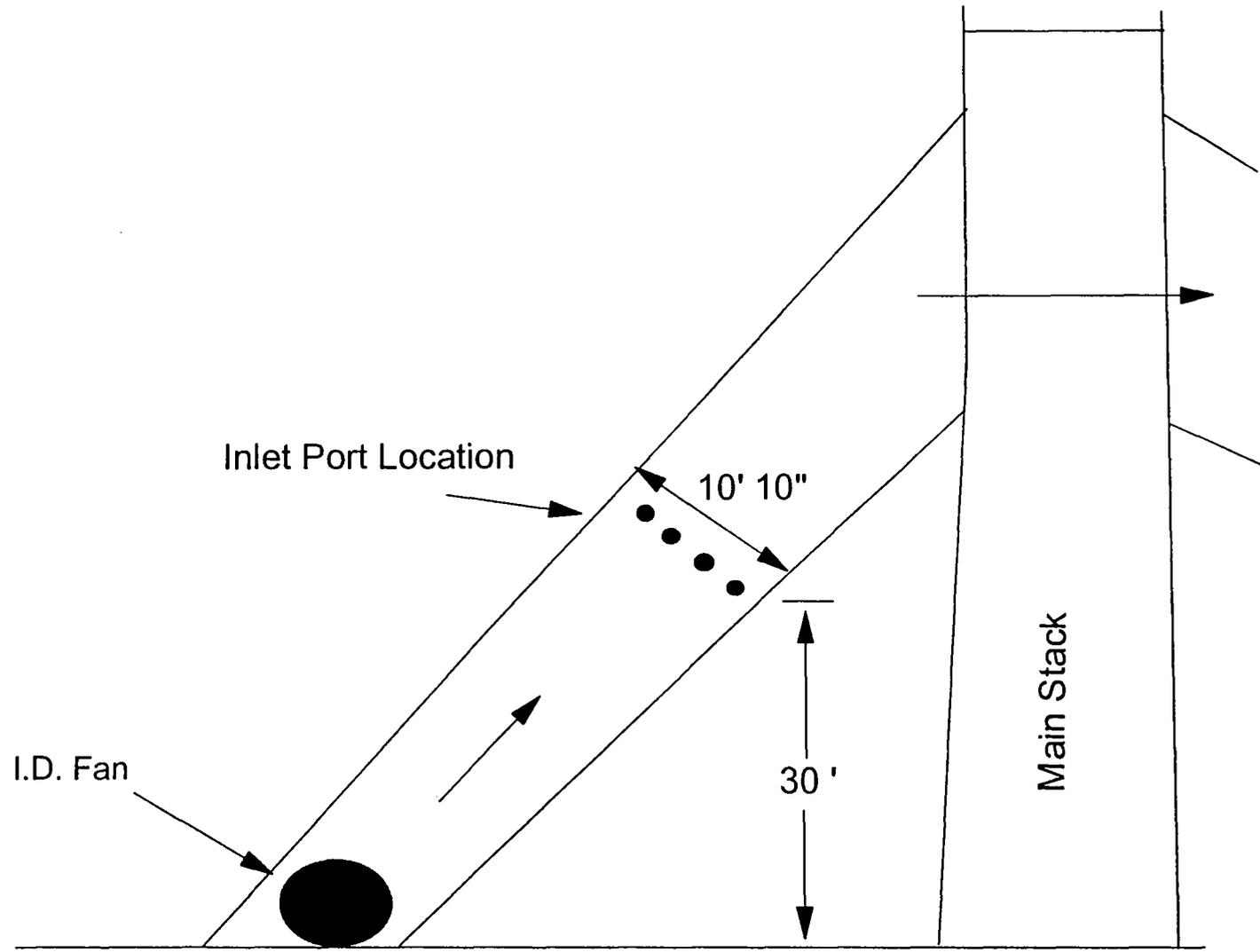


Figure 4-1. Venturi Inlet Sampling Location

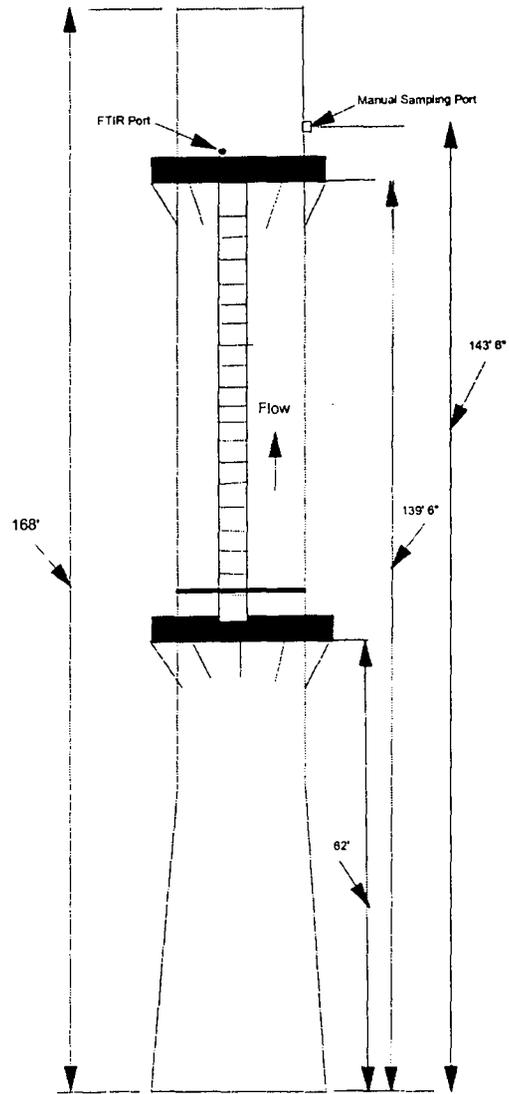


Figure 4-2. Venturi Outlet Sampling Location

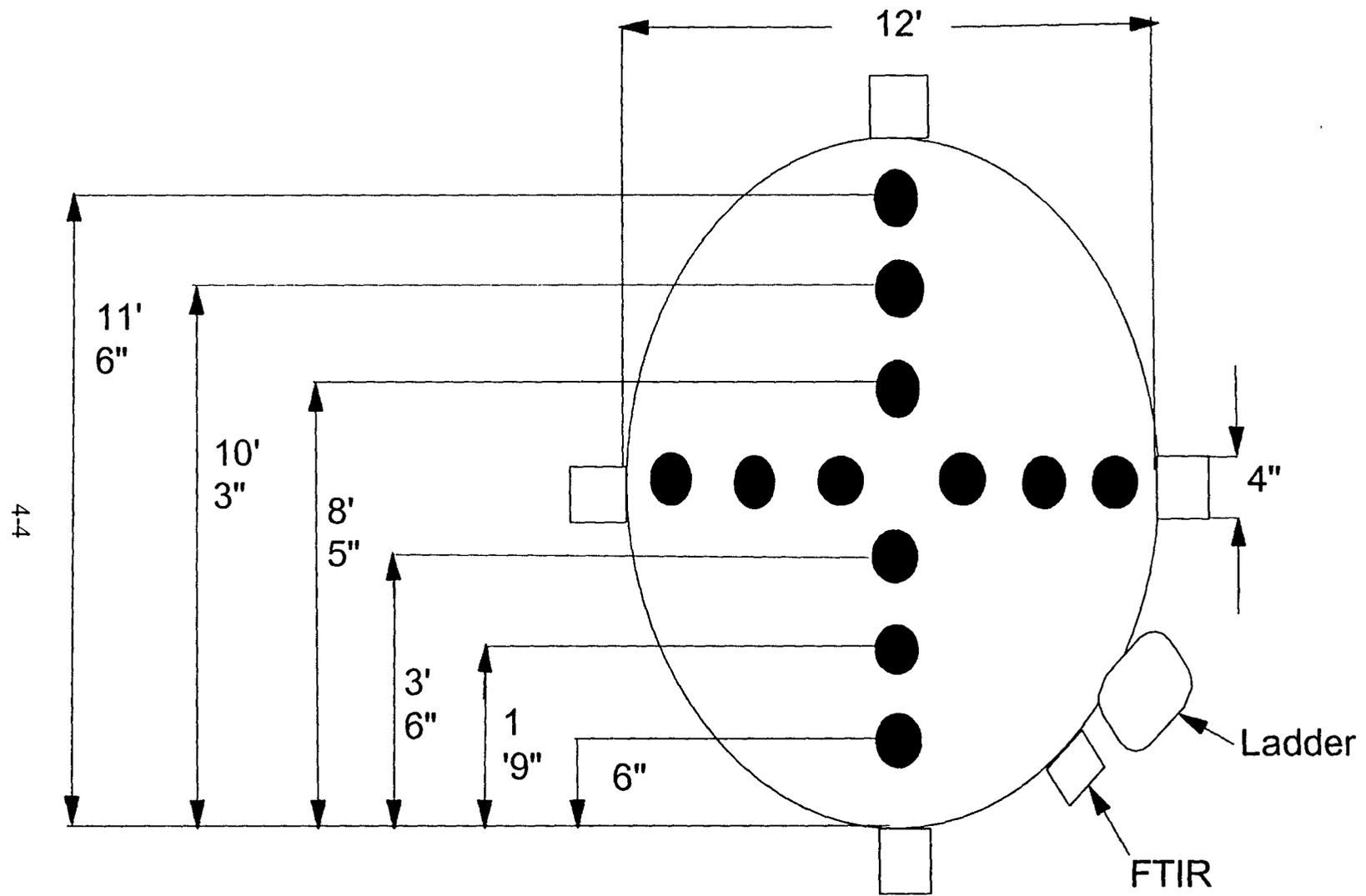
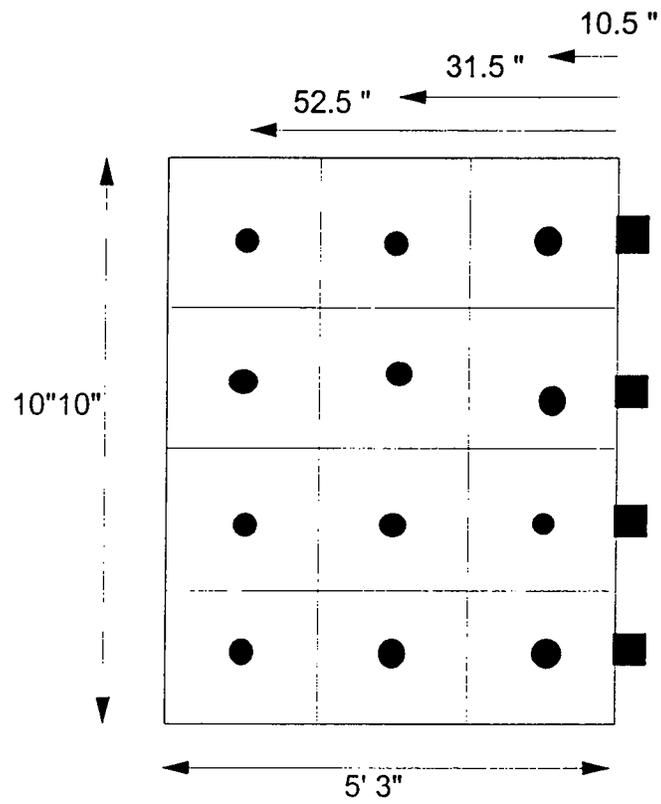


Figure 4-3. Outlet Traverse Point Layout



**Figure 4-4. Inlet Traverse Point Layout**



## **5.0 SAMPLING AND ANALYTICAL PROCEDURES BY ANALYTE**

The sampling and analytical procedures used for the sintering plant test program are the most recent revisions of the published EPA methods. In this section, descriptions of each sampling and analytical method by analyte are provided.

### **5.1 Particulate Matter and Metals Emissions Testing Using EPA Method 29**

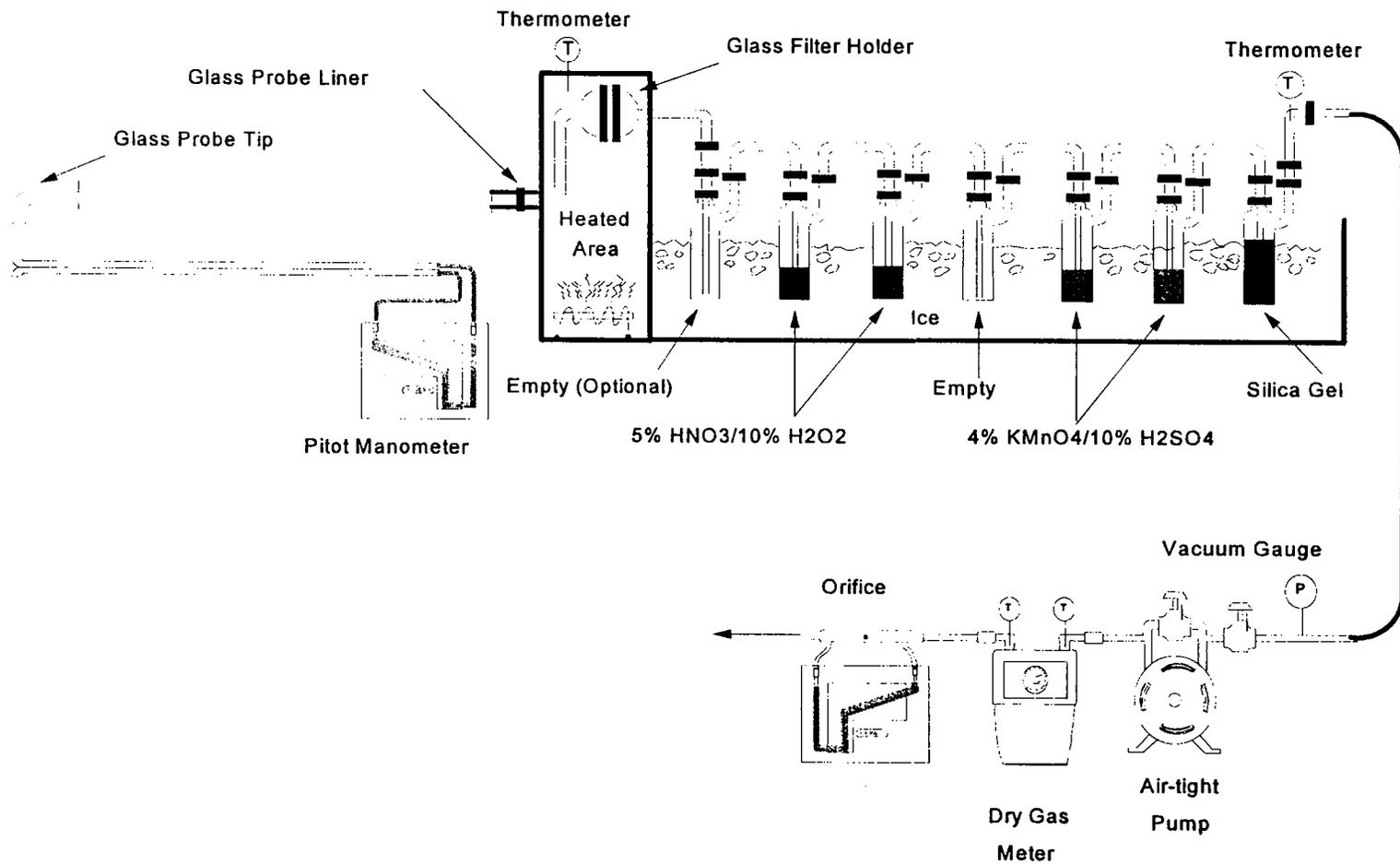
Sampling for Particulate Matter (PM) and metals was performed according to the EPA Method 29 protocol. This method is applicable to the determination of particulate mass and Pb, Ni, Cr, Mn, Se, Be, Sb, Co, Cd, As, and Hg emissions from various types of process controls and combustion sources. Analyses of the test samples were performed for the metals listed employing inductively-coupled argon plasma spectroscopy (ICAPS) and cold vapor atomic absorption (CVAA) for mercury instrumental measurements. Mercury was analyzed using EPA Method 7470A.

PM emissions were also determined from this sampling train. Particulate concentrations are based on the weight gain of the filter and the front half acetone rinses (probe, nozzle, and filter holder). The procedures which were used to determine particulate concentrations from the Method 29 samples may have resulted in some mercury losses due to volatilization during sample workup for PM determination. After the gravimetric analyses were completed, the sample fractions were then analyzed for the target metals as discussed in Section 5.1.6.

#### **5.1.1 Method 29 Sampling Equipment**

The Method 29 methodology uses the sampling train shown in Figure 5-1. The 7-impinger train consists of a borosilicate glass nozzle/probe liner followed by a heated filter

Figure 5-1. EPA Method 29 Sampling Train



assembly with a Teflon® filter support, a series of impingers and the usual EPA Method 5 meterbox and vacuum pump. The sample was not exposed to any metal surfaces in this train.

The contents of the sequential impingers were:

- An empty knockout impinger is the first impinger;
- Two impingers with a 5% nitric acid (HNO<sub>3</sub>)/10% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) solution;
- An empty knockout impinger;
- Two impingers with a 4% potassium permanganate (KMnO<sub>4</sub>)/10% sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) solution; and
- An impinger containing indicating silica gel.

## **5.1.2 Method 29 Sampling Equipment Preparation**

### **5.1.2.1 Glassware Preparation**

Glassware was washed in soapy water, rinsed with hot tap water, soaked in 10% HNO<sub>3</sub> for 12 hours, rinsed with Type II water, and then rinsed with acetone. This procedure included all the glass components of the sampling train including the glass nozzles plus any sample bottles, erlenmeyer flasks, petri dishes, graduated cylinders or stirring rods that are used during recovery. Non-glass components (such as the Teflon®-coated filter screens and seals, tweezers, Teflon® squeeze bottles, Nylon® probe brushes and Nylon® nozzle brushes) were cleaned following the same procedure. The specifics of the cleaning procedure are presented in Table 5-1.

**Table 5-1. Glassware Cleaning Procedure (Train Components)**

NOTE: USE DISPOSABLE GLOVES AND ADEQUATE VENTILATION

1. Soak all glassware in hot soapy water (Alconox®).
2. Tap water rinse to remove soap.
3. Distilled/deionized H<sub>2</sub>O rinse (X3).<sup>a</sup>
4. Soak in 10% HNO<sub>3</sub> solution for 12 hours.
5. Distilled/Deionized H<sub>2</sub>O rinse (X3).
6. Acetone (X3).
7. Cap glassware with clean glass plugs or Parafilm®.
8. Mark cleaned glassware with color-coded identification sticker.

<sup>a</sup>(X3) = Three Times.

#### 5.1.2.2 Reagent Preparation

The sample train filters were Pallflex Tissuequartz 2500 QAS filters. The acids and hydrogen peroxide were Baker “Intra-analyzed” grade or equivalent. The peroxide was purchased specifically for this test site.

The reagent water was Baker “Analyzed” low metals grade or equivalent. The lot number, manufacturer and grade of each reagent that is used is recorded in the laboratory notebook.

The HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> and KMnO<sub>4</sub>/H<sub>2</sub>SO<sub>4</sub> solutions were prepared daily immediately prior to sampling according to Section 4.2.1 of the reference method. The analyst wears both safety glasses and protective gloves when the reagents are mixed and handled. Each reagent has its own designated transfer and dilution glassware. This glassware was marked for identification with a felt tip glass marking pen and used only for the reagent for which it was designated.

### 5.1.2.3 Equipment Preparation

The remaining preparation included calibration and leak checking of the all the train equipment, including meterboxes, thermocouples, nozzles, pitot tubes, and umbilicals. Referenced calibration procedures were followed when available, and the results properly documented and retained. A discussion of the techniques used to calibrate this equipment is presented below.

**Type-S Pitot Tube Calibration.** The EPA has specified guidelines concerning the construction and geometry of an acceptable Type-S pitot tube. If the specified design and construction guidelines are met, a pitot tube coefficient of 0.84 is used. Information pertaining to the design and construction of the Type-S pitot tube is presented in detail in Section 3.1.1 of EPA Document 600/4-77-027b. Only Type-S pitot tubes meeting the required EPA specifications are used. Pitot tubes are inspected and documented as meeting EPA specifications prior to field sampling.

**Sampling Nozzle Calibration.** Glass nozzles were used for isokinetic sampling. Calculation of the isokinetic sampling rate requires that the cross sectional area of the sampling nozzle be accurately and precisely known. All nozzles were thoroughly cleaned, visually inspected and calibrated according to the procedure outlined in Section 3.4.2 of EPA Document 600/4-77-027b.

**Temperature Measuring Device Calibration.** Accurate temperature measurements are required during source sampling. Bimetallic stem thermometers and thermocouple temperature sensors are calibrated using the procedure described in Section 3.4.2 of EPA document 600/4-77-027b. Each temperature sensor was calibrated at a minimum of two points over the anticipated range of use against a NBS-traceable mercury-in-glass thermometer. All sensors were calibrated prior to field sampling.

**Dry Gas Meter Device Calibration.** Dry gas meters (DGMs) are used in the Method 29 sampling trains to monitor the sampling rate and to measure the sample volume. All DGMs were calibrated to document the volume correction factor just prior to shipping of the equipment to the field. Post-test calibration checks were performed as soon as possible after the equipment was returned to the ERG Laboratory. Pre- and post-test calibrations should agree to within 5%.

Prior to calibration, a positive pressure leak check of the system was performed using the procedure outlined in Section 3.3.2 of EPA document 600/4-77-237b. The system was placed under approximately 10 inches of water pressure and a gauge oil manometer is used to determine if a pressure decrease could be detected over a one-minute period. If leaks were detected, they were eliminated before actual calibrations were performed.

After the sampling console was assembled and leak checked, the pump was to run for 15 minutes, to allow the pump and DGM to warm up. The valve was then adjusted to obtain the desired flow rate. For the pre-test calibrations, data were collected at orifice manometer settings ( $\Delta H$ ) of 0.5, 1.0, 1.5, 2.0, 3.0 and 4.0 in  $H_2O$ . Gas volumes of  $5 \text{ ft}^3$  were used for the two lower orifice settings, and volumes of  $10 \text{ ft}^3$  are used for the higher settings. The individual gas meter correction factors ( $Y_i$ ) were calculated for each orifice setting and averaged. The method requires that each of the individual correction factors fall within  $\pm 2\%$  of the average correction factor or the meter is cleaned, adjusted, and recalibrated. In addition, ERG requires that the average correction factor be within  $1.00 \pm 1\%$ . For the post-test calibration, the meter was calibrated three times at the average orifice setting and vacuum which were used during the actual test.

### **5.1.3 Method 29 Sampling Operations**

#### **5.1.3.1 Preliminary Measurements**

Prior to sampling, preliminary measurements are required to ensure isokinetic sampling. These preliminary measurements include determining the traverse point locations and performing a preliminary velocity traverse and a cyclonic flow check. These measurements were used to calculate a "K factor." The K factor was used to determine an isokinetic sampling rate from stack gas flow readings taken during sampling.

Measurements were then made of the duct inside diameter, port nozzle length, and the distances to the nearest upstream and downstream flow disturbances. These measurements were then used to determine sampling point locations by following EPA Reference Method 1 guidelines. The distances were then marked on the sampling probe using an indelible marker.

#### **5.1.3.2 Assembling the Train**

The assembly of the Method 29 sampling train components was completed in the recovery trailer and final train assembly was performed at the stack location. First, the empty, clean impingers were assembled and laid out in the proper order in the recovery trailer. Each ground glass joint was carefully inspected for hairline cracks. After the impingers were loaded, each impinger was weighed, and the initial weight and contents of each impinger were recorded on a recovery data sheet. The impingers were connected together using clean glass U-tube connectors and arranged in the impinger bucket. The height of all the impingers was approximately the same to obtain a leak free seal. The open ends of the train were sealed with Parafilm® or clean ground glass caps.

The filter was loaded into the filter holder in the recovery trailer. The filter holder was then capped off and placed in the impinger bucket. To avoid contamination of the sample,

sealing greases were not used. The train components were transferred to the sampling location and assembled as previously shown in Figure 5-1.

### 5.1.3.3 Sampling Procedures

After the train was assembled, the heaters for the probe liner and heated filter box were turned on. When the system reaches the appropriate temperatures, the sampling train was ready for pre-test leak checking. The filter temperature was maintained at  $120 \pm 14^{\circ}\text{C}$  ( $248 \pm 25^{\circ}\text{F}$ ). The probe temperature was maintained above  $100^{\circ}\text{C}$  ( $212^{\circ}\text{F}$ ).

The sampling trains were leak checked at the start and finish of sampling. (Method 5 protocol requires post-test leak checks and recommends pre-test leak checks.) ERG protocol also incorporates leak checks before and after every port change. An acceptable pre-test leak rate is less than 0.02 acfm ( $\text{ft}^3/\text{min}$ ) at approximately 15 inches of mercury (in. Hg). If during testing, a piece of glassware needed to be emptied or replaced, a leak check was performed before the glassware piece was removed, and after the train was re-assembled.

To leak check the assembled train, the nozzle end was capped off and a vacuum of 15 in. Hg was pulled in the system. When the system was evacuated, the volume of gas flowing through the system was timed for 60 seconds. After the leak rate was determined, the cap was slowly removed from the nozzle end until the vacuum dropped off, and then the pump was turned off. If the leak rate requirement was not met, the train was systematically checked by first capping the train at the filter, at the first impinger, etc., until the leak was located and corrected.

After a successful pre-test leak check was conducted, all train components were at their specified temperatures and initial data were recorded [dry gas meter (DGM) reading], the test was initiated. Sampling train data were recorded periodically (specific interval to be determined) on standard data forms. A checklist for sampling is included in Table 5-2.

## Table 5-2. Sampling Checklist

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### Before Test Starts:

1. Check impinger set (right order and number). Verify probe markings, and re-mark if necessary.
  2. Check that you have all the correct pieces of glassware.
  3. Check for data sheets and barometric pressure.
  4. Bag sampling equipment needs to be ready for Method 3 analysis.
  5. Leak check pitot tubes.
  6. Examine meter box - level it and confirm that the pump is operational.
  7. Assemble train to the filter and leak check at 15 in. Hg. Attach probe to train and do final leak check; record leak rate and pressure on sampling log.
  8. Check out thermocouples - make sure they are reading correctly.
  9. Turn on heaters and check to see that their temperatures are increasing.
  10. Check that cooling water is on and flowing. Add ice to impinger buckets.
  11. Check isokinetic K-factor - make sure it is correct. (Refer to previous results to confirm assumptions. Two people should calculate the K-factor independently to double check it).
  12. Have a spare probe liner, probe sheath, meter box and filter ready to go at location.
-

## Table 5-2. Continued

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### During Test:

1. Notify crew chief of any sampling problems ASAP. Train operator should fill in sampling log.
2. Perform simultaneous/concurrent testing with other locations (if applicable). Maintain filter temperature between  $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$ . Keep temperature as steady as possible. Maintain impinger temperatures below  $68^{\circ}\text{F}$ . Maintain probe temperature above  $212^{\circ}\text{F}$ .
3. Leak check between ports and record on sampling log.
4. Record sampling rate times and location for the fixed gas ( $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{O}_2$ ) sample (if applicable).
5. Blow back pitot tubes at inlet location every 15 minutes.
6. Change filter if pressure drop exceeds 20 in. Hg.
7. Check permanganate impinger solutions every 1/2 hr for reagent depletion.
8. Check impinger silica gel every 1/2 hr; if indicator disappears request a pre-filled impinger from van lab and replace.
9. Check manometer fluid levels and zero every hour.

### After Test is Completed:

1. Record final meter reading.
  2. Check completeness of data sheet.
  3. Do final leak check of sampling train at maximum vacuum during test.
  4. Leak check each leg of pitot tubes.
-

**Table 5-2. Continued**

- 
5. Disassemble train. Cap sections. Take sections to recovery trailer.
  
  6. Probe/cyclone recovery (use 500 mL bottles)
    - a) For acetone rinses (all trains)
      - Attach flask to end of probe
      - Add 50 mL of acetone
      - Put a brush down probe, and brush back and forth
      - Rinse back and forth in probe
      - Empty out acetone in sample jar
      - Do this 3 times so that the final combined acetone rinse volume is  $\leq 150$  mL.
  
    - b)
      - Rinse nozzle and probe 3X with 0.1N HNO<sub>3</sub>
      - Collect approximately 100 mL of rinse into sampling jar.
  
  7. Reattach nozzle and cap for next day, store in dry safe place.
  
  8. Make sure data sheets are completely filled out and give to location leader.
- 

The leak rates and sampling start and stop times were recorded on the sampling task log. Also, any other events that occur during sampling were recorded on the task log, such as pitot cleaning, thermocouple malfunctions, heater malfunctions, or any other unusual occurrences.

At the conclusion of the test run, the sample pump (or flow) was turned off, the probe was removed from the duct, a final DGM reading was taken, and a post-test leak check was completed. The procedure was identical to the pre-test procedure; however, the vacuum should be at least one inch Hg higher than the highest vacuum attained during sampling. An acceptable leak rate is less than 4% of the average sample rate or 0.02 acfm (whichever is lower). If a final leak rate on-site did not meet the acceptance criterion, the test run may still be accepted upon

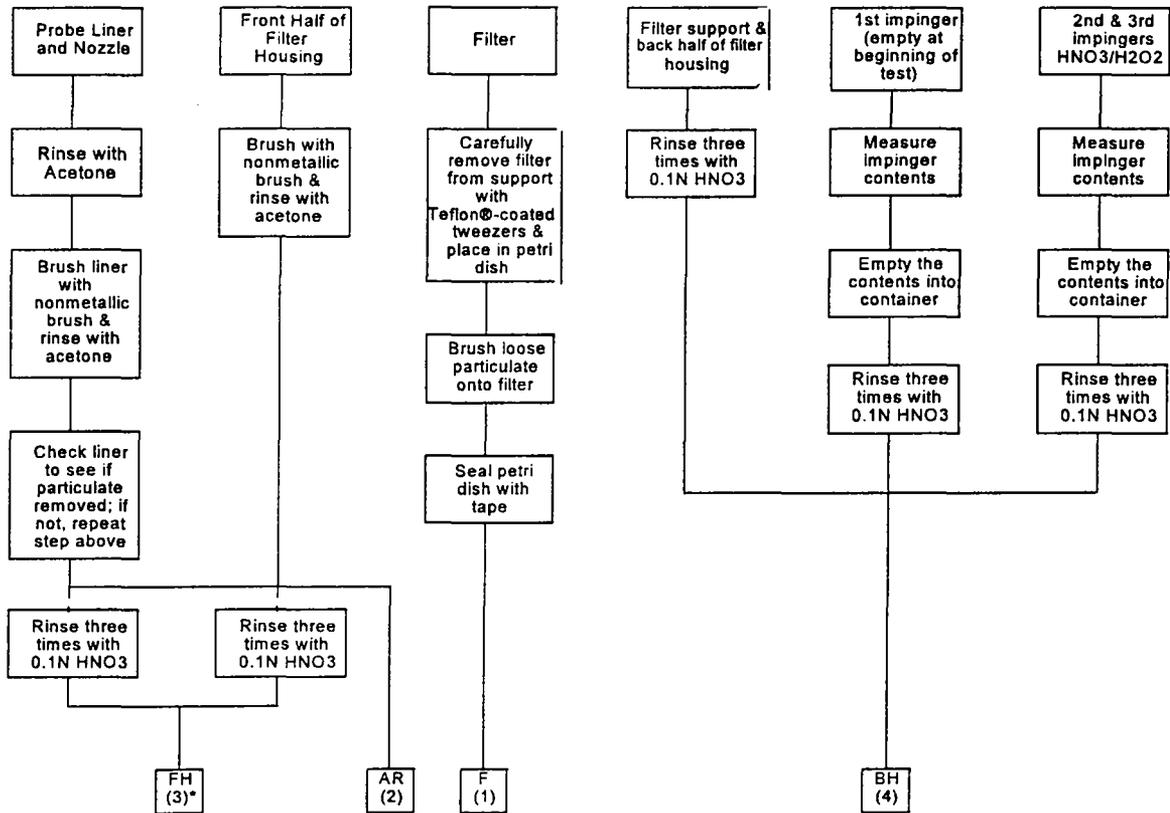
approval of the EPA test administrator. If so, the measured leak rate was reduced by subtracting the allowable leak rate from it and then multiplied for the period of time in which the leak occurred. This “leaked volume” is then subtracted from the measured gas volume in order to determine the final gas sample volume.

#### **5.1.4 Method 29 Sample Recovery**

Recovery procedures begin as soon as the probe was removed from the stack and the post-test leak check was completed.

To facilitate transfer from the sampling location to the recovery trailer, the sampling train was disassembled into three sections: the nozzle/probe liner, filter holder and impingers in their bucket. Each of these sections was capped with Teflon® tape or Parafilm® before removal to the recovery trailer. All train components were rinsed and the samples collected in separate, pre-labeled, pre-cleaned sample containers to avoid cross contamination of inlet and outlet samples.

Once in the trailers, the sampling train was recovered as separate front and back half fractions. A diagram illustrating front half and back half sample recovery procedures is shown in Figure 5-2. No equipment with exposed metal surfaces was used in the sample recovery procedures. The weight gain in each of the impingers was recorded to determine the moisture content in the flue gas. Following weighing of the impingers, the front half of the train was recovered, which included the filter and all sample-exposed surfaces forward of the filter. The probe liner was rinsed with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces were wetted. The acetone was quantitatively collected into the appropriate bottle. This rinse was followed by additional brush/rinse procedures using a non-metallic brush; the probe was held in an inclined position and acetone was squirted into the upper end as the brush was pushed through with a twisting action. All of the acetone and particulate was caught in the sample container. This procedure was repeated until no visible



\* Number in parentheses indicates container number

Figure 5-2. Method 29 Sample Recovery Scheme

particulate remains and finished with a final acetone rinse of the probe and brush. The front half of the filter was also rinsed with acetone until all visible particulate was removed. After all front half acetone washes were collected, the cap was tightened, the liquid level marked and the bottle weighed to determine the acetone rinse volume. The method specifies that a total of 100 mL of acetone must be used for rinsing these components. For blank correction purposes, the exact weight or volume of acetone used was measured. An acetone reagent blank of approximately the same volume as the acetone rinses was analyzed with the samples.

The nozzle/probe liner and front half of the filter holder were rinsed three times with 0.1N HNO<sub>3</sub> and placed into a separate amber bottle. Cap tightly, record the weight of the combined rinse and mark the liquid level. The filter was placed in a clean, well-marked glass petri dish (Container 1) and sealed with Teflon® tape. Approximately 100 mL of this rinse was required.

Prior to recovering the back half impingers, the contents were weighed for moisture content determinations. Any unusual appearance of the filter or impinger contents was noted.

The contents in the knockout impinger (if used) were recovered into a pre-weighed, pre-labeled bottle with the contents from the HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> impingers (Container 4). These impingers and connecting glassware were rinsed thoroughly with 0.1N HNO<sub>3</sub>, the rinse was captured in the impinger contents bottle, and a final weight was taken. Again, the method specifies a total of 100 mL of 0.1N HNO<sub>3</sub> be used to rinse these components. The weight of reagent used for rinsing was determined by weighing the impinger contents bottle before and after rinsing the glassware. A nitric acid reagent blank of approximately the same volume as the rinse volume was analyzed with the samples. The acidified permanganate impinger solutions were combined into a single sample container. Any residue from the impingers was recovered with 25 mL of 8N HCl solution and was collected in a separate container.

After final weighing, the silica gel from the train was saved for regeneration. The ground glass fittings on the silica gel impinger were wiped off after sample recovery to assure a leak tight fit for the next test.

A reagent blank was recovered in the field for each of the following reagents;

- Acetone blank - 100 mL sample size;
- 0.1N nitric acid blank - 300 mL sample size;
- 5% nitric acid/10% hydrogen peroxide blank - 200 mL sample size;
- Acidified potassium permanganate blank - 200 mL sample size (this blank should have a vented cap);
- 8N hydrochloric acid blank - 225 mL sample size (25 mL 8N HCl plus 200 mL water);
- Dilution water - 200 mL sample size; and
- Filter blank - one each.

Each reagent blank was of the same lot as was used during the sampling program. The volumes collected were greater than required for sample preparation in order to provide sufficient amounts in case of sample loss during preparation or to compensate for larger volumes of train rinses. Each lot number and reagent grade were recorded on the field blank label. One field blank was collected using an on-site sampling train. One glassware proof blank was collected for each train prior to sampling.

The liquid level of each sample container was marked on the bottle in order to determine if any sample loss occurred during shipment. If sample loss occurred, the sample may be voided or a method may be used to incorporate a correction factor to scale the final results depending on the volume of the loss.

Cold Vapor Atomic Absorption Spectroscopy (CVAAS) was used to analyze for mercury by EPA method 7470A. The detection limits of the individual metals are dependent on the detection limit of the analytical method, the volume of the aqueous sample presented for analysis and the total volume of gaseous sample collected in the sampling trains. Following the protocol of Method 29, the fractions that were collected for analysis from each train were:

- Fraction 1--Filter;
- Fraction 2--Probe and filter front half acetone rinses;
- Fraction 3--Probe and filter front half acid rinse;
- Fraction 4--Impingers 1-3 contents and acid rinse of impingers and filter back half;
- Fraction 5a--Impinger 4 contents and 100 mL 0.1N nitric acid rinse;
- Fraction 5b--Impinger 5-6 contents plus 100 mL permanganate and 100 mL water rinses; and
- Fraction 5c--25 mL 8N HCl acid rinse and water rinses of impingers 5-6, place in containers with 200 mL water.

After sample preparation, Fractions 1-3 were combined for analysis for all target analytes (an aliquot is removed for Hg). Fractions 4, 5a, 5b and 5c were analyzed individually after preparation. Fraction 4 was analyzed for all analytes (aliquot for Hg removed). Fractions 5a, 5b, and 5c were analyzed for Hg only. Since there were multiple fractions to be analyzed (5 for Hg and 2 for other metals) the method detection limit (MDL) is the sum of the individual detection limits for each fraction analyzed. For Hg this will increase the MDL over that seen for Method 101a where the permanganate is the only collection medium. Using an instrumental detection limit (IDL) for cold vapor atomic absorption (CVAA) and inductively coupled argon plasma (ICAP), Table 5-3 gives the total detectable amounts that were possible.

The method detection limits for the various metals of interest are summarized in Table 5-4. The sampling flow rate at the inlet and outlet locations were dictated by the flow rate

**Table 5-3. Analytical Detection Limits**

Metal	IDL μg/mL	Analysis Fraction					Total Detectable Amount μg
		1, μg	2, μg	3, μg	4, μg	5, μg	
Hg	0.0002	0.4	0.6	0.2	0.6	1 μg	2.8
As	0.005	1.0	1.13	NA	NA	NA	2.1
Be	0.001	0.2	0.23	NA	NA	NA	0.43
Cd	0.001	0.2	0.23	NA	NA	NA	0.43
Cr	0.002	0.4	0.45	NA	NA	NA	0.85
Pb	0.002	0.4	0.45	NA	NA	NA	0.85
Sb	0.004	0.8	0.9	NA	NA	NA	1.7
Co	0.001	0.2	0.225	NA	NA	NA	0.43
Mn	0.002	0.4	0.45	NA	NA	NA	0.85
Ni	0.003	0.6	0.68	NA	NA	NA	1.28
Se	0.003	0.6	0.68	NA	NA	NA	1.28

Note: Hg analysis by CVAA Method 7470A, all others by Method 6010A (ICAPS). CVAA assumes an analysis volume of 10 mL. NA = Not applicable.

of the stack gas since isokinetic sampling was performed at these locations. The nominal sample time and flow rate selected by the EPA Work Assignment Manager are presented in Table 5-4 along with the associated method detection limits.

### **5.1.5 Particulate Analysis**

The same general gravimetric procedure described in Method 5, Section 4.3, was followed. Both filters and precleaned beakers were weighed to a constant weight before use in the field. The same balance used for taring was used for weighing the samples.

The acetone rinses were evaporated to dryness under a clean hood at 20°C (68°F) in a tared beaker. The residue was desiccated for 24 hours in a desiccator containing fresh room temperature silica gel. The filter was also desiccated under the same conditions to a constant weight. Weight gain was reported to the nearest 0.1 mg. Each replicate weighing must agree to within 0.5 mg or 1% of total weight less tare weight, whichever is greater, between two consecutive weighings, and must be at least 6 hours apart. The balance room was temperature and humidity controlled. The filter tare and final weights will be determined under the same conditions.

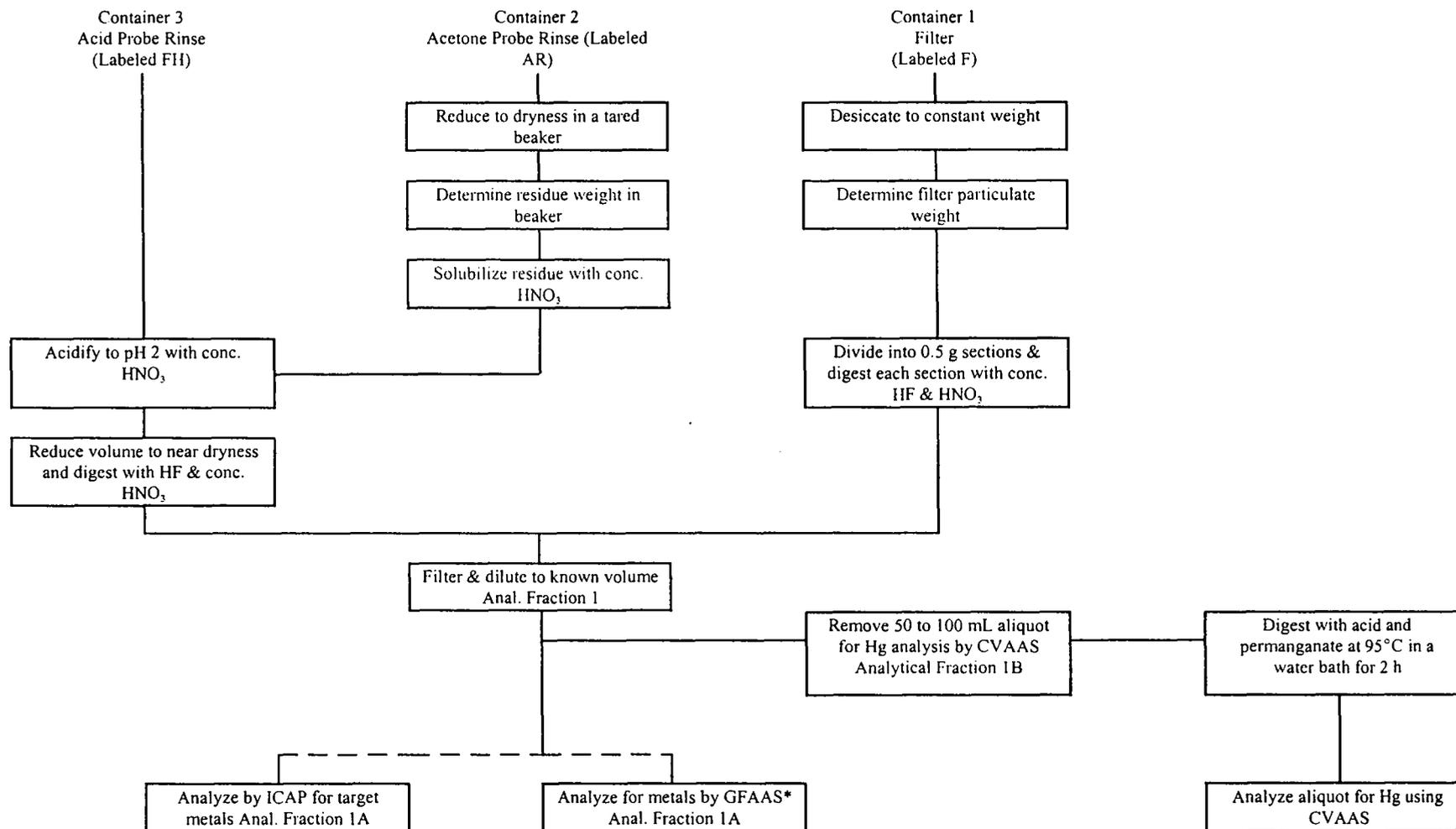
### **5.1.6 Metals Analytical Procedures**

A diagram illustrating the sample preparation and analytical procedures for the target metals is shown in Figure 5-3.

The acetone probe rinse (container No. 2) was allowed to reduce to dryness in a tared beaker and any residue was weighed to a constant weight. This residue was then solubilized with concentrated nitric acid and this solution was added to the nitric acid rinse of the probe (Container No. 3). This combined solution was then acidified to a pH of 2 with concentrated

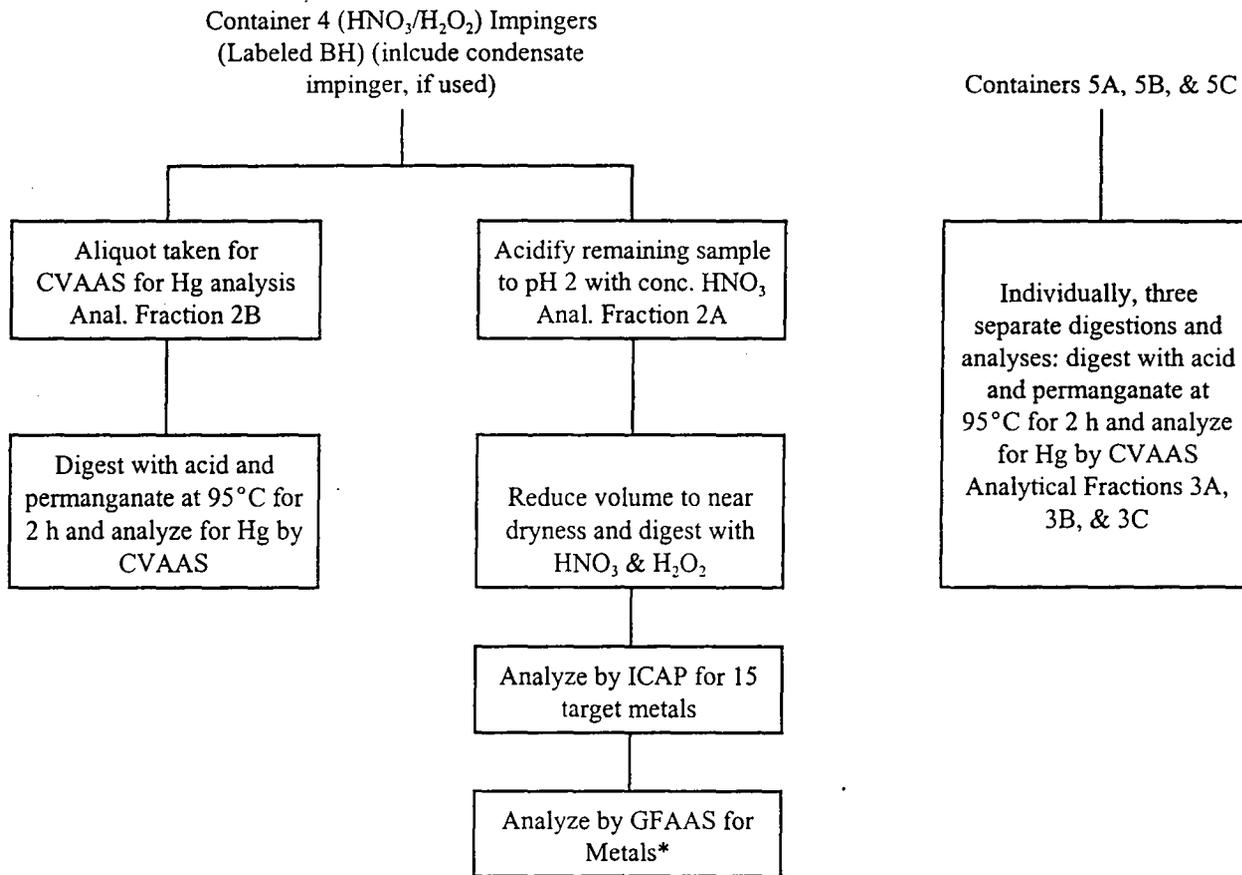
**Table 5-4. Method 29 Detection Limits**

Sampling Time, hours	4
Sampling Rate, cfm	0.75
Sampling Volume, cubic meters	5.1
	<b>MDL, <math>\mu\text{g}/\text{m}^3</math></b>
Hg	0.55
As	0.41
Be	0.08
Cd	0.08
Cr	0.17
Pb	0.17
Sb	0.33
Co	0.08
Mn	0.17
Ni	0.25
Se	0.25



\*Analyze by AAS for metals found at less than 2 µg/mL in digestate solution, if desired. Or analyze for each metal by AAS, if desired.

**Figure 5-3. Method 29 Sample Preparation and Analysis Scheme**



\*Analysis by AAS for metals found at less than 2 µg/mL in digestate solution, if desired. Or analyzed for each metal by AAS, if desired.

**Figure 5-3. Continued**

nitric acid, the volume reduced to near dryness and digested with concentrated nitric and hydrofluoric acids in a microwave pressure vessel.

The filter (Container No. 1) was weighed to a constant weight and then divided into 0.5 g sections and digested with concentrated nitric and hydrofluoric acids in a microwave pressure vessel. The microwave digestion took place over a period of 10 to 15 minutes in intervals of 1 to 2 minutes at 600 watts. Both the digested filter and the digested probe rinses were combined, filtered and brought to a known volume (nominally 200 mL). This analysis fraction was then divided for analysis by CVAA for Hg (following additional digestion) and by ICAP for the other target metals.

An aliquot from the contents of container No. 4 (nitric acid/peroxide impinger absorbing solution) was removed and digested following the procedures given in Method 29 and then analyzed for Hg by CVAA. The remaining volume was acidified to pH 2, the volume reduced to near dryness and digested in a microwave as discussed above. After bringing the digestate to a known volume, the solution was analyzed by ICAP for the remaining target metals.

The contents of containers 5A, 5B and 5C were digested separately by the procedures given in Method 29 and then analyzed for Hg by CVAA.

A total of two (2) fractions were analyzed for all target metals except Hg by Method 6010A and a total of five (5) fractions were analyzed for Hg by Method 7470A.

#### **5.1.7 Quality Control for Metals Analytical Procedures**

All quality control procedures specified in the test method were followed. All field reagent blanks were processed, digested and analyzed as specified in the test method. For optimum sensitivity in measurements, the concentrations of target metals in the solutions should be at least 10 times analytical detection limits.

#### 5.1.7.1 ICAP Standards and Quality Control Samples

The quality control procedures included running two standards for instrument checks (or frequency of 10%), two calibration blank runs (or frequency of 10%), one interference check sample at the beginning of the analysis (must be within 10% or analyze by standard addition), one quality control sample to check the accuracy of the calibration standards (must be within 10% of calibration), one duplicate analysis and one standard addition for every 10 samples (must be within 5% of average or repeat all analysis).

Standards less than 1  $\mu\text{g/mL}$  of a metal were prepared daily; those with concentrations greater than this were made weekly or bi-monthly.

All samples were analyzed in duplicate. A matrix spike on one front half sample and one back half for each 10 field samples was analyzed. If recoveries of less than 75% or greater than 120% were obtained for the matrix spike, each sample was analyzed by the method of additions. One quality control sample was analyzed to check the accuracy of the calibration standards. The results must be within 10% or the calibration will be repeated.

#### 5.1.7.2 Cold Vapor Atomic Absorption Standards and Quality Control Samples

A 10 $\mu\text{g/mL}$  intermediate Hg standard was prepared fresh weekly. A fresh daily 200 Hg/mL Hg working standard was also prepared. At least five separate aliquots of the working Hg standard solution and a blank were used to prepare the standard curve. Quality control samples were prepared by making a separate 10 $\mu\text{g/mL}$  standard and diluting it until the control sample is within the calibration range. These procedures assessed the quality control of the analysis, but do not address the potential negative bias due to Hg losses from the filter due to volatilization.

## **5.2 CDD/CDF and PAH Emissions Testing Using EPA Method 23**

The sampling and analytical method for determining flue gas emissions of Polychlorinated Dibenzo-*p*-Dioxins and Polychlorinated Dibenzofurans (CDD/CDF) is EPA Method 23. Samples collected with this method were also analyzed for Polycyclic Aromatic Hydrocarbons (PAHs) emissions.

### **5.2.1 Method 23 Sampling Equipment**

The method uses the sampling train shown in Figure 5-4. Basically, the sampling system is similar to a Method 5 train with the following exceptions:

- All components (glass probe/nozzle liner, all other glassware, filters) are pre-cleaned using solvent rinses and extraction techniques; and
- A condensing coil and XAD-2® resin absorption module are located between the filter and impinger train.

All sampling equipment specifications are detailed in the reference method.

### **5.2.2 Method 23 Equipment Preparation**

In addition to the standard EPA Method 5 requirements, Method 23 includes several unique preparation steps which ensure that the sampling train components are not contaminated with organics that may interfere with analysis. The glassware, glass fiber filters and absorbing resin were cleaned and the filters and resin were checked for 42 residuals before they were packed.

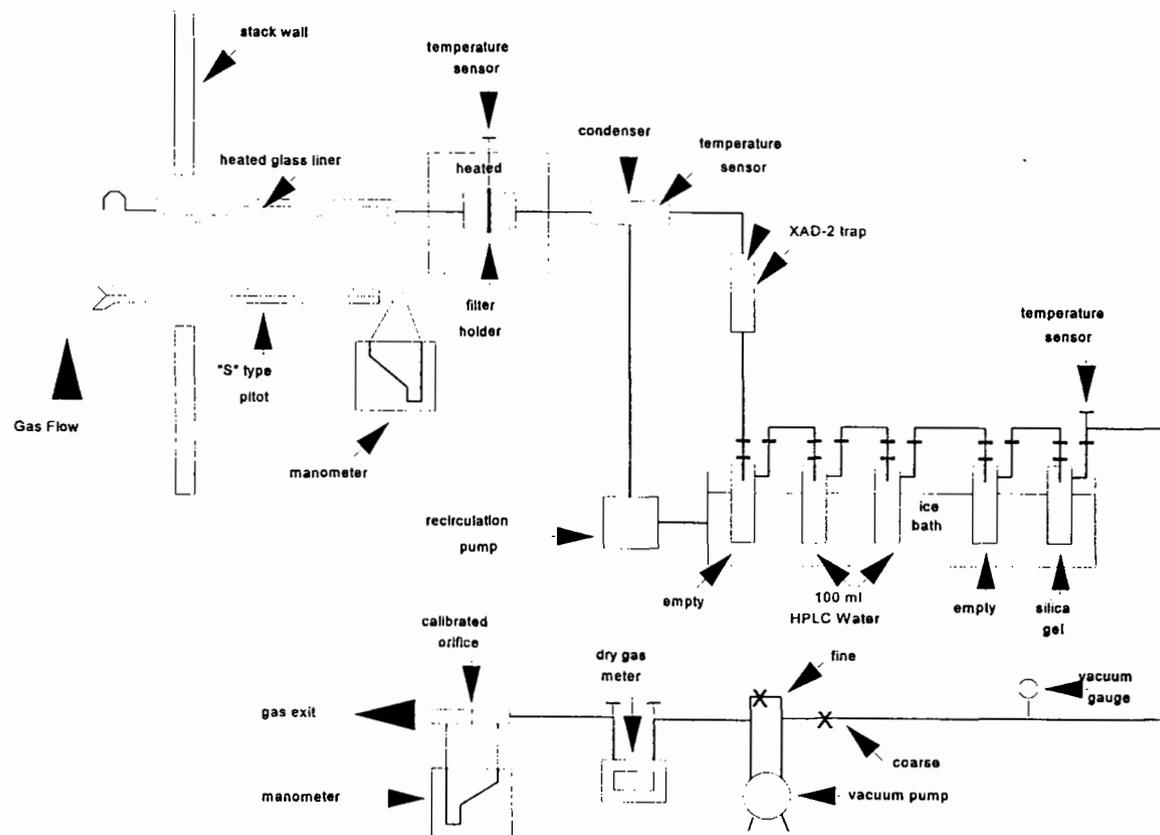


Figure 5-4. Method 23 Sampling Train Configuration

### 5.2.2.1 Glassware Preparation

Glassware was cleaned as shown in Table 5-5. Glassware was washed in soapy water, rinsed with distilled water, baked and then rinsed with acetone followed by methylene chloride. Clean glassware was allowed to dry under a hood loosely covered with foil to prevent laboratory contamination. Once the glassware was dry, the air exposed ends were sealed with methylene chloride rinsed aluminum foil. All the glass components of the sampling train including the glass nozzles plus any flasks, petri dishes, graduated cylinders and pipets that are used during sampling and recovery were cleaned according to this procedure. Non-glass components (such as the Teflon®-coated filter screens and seals, tweezers, Teflon® squeeze bottles, Nylon® probe brushes and Nylon® nozzle brushes) were cleaned following the same procedure except that no baking was performed.

### 5.2.2.2 XAD-2® Resin and Filters Preparation

XAD-2® absorbing resin and glass fiber filters were pre-cleaned by separate procedures according to the specified method. Only pesticide grade solvents and HPLC grade water were used to prepare for organic sampling, and to recover these samples. The lot number, manufacturer and grade of each reagent used were recorded in the laboratory notebook.

To prepare the filters, a batch of 50 was placed in a Soxhlet extractor pre-cleaned by extraction with toluene. The Soxhlet was charged with fresh toluene and refluxed for 16 hours. After the extraction, the toluene was analyzed as described in Sections 5.2 and 5.3 of the reference method for the presence of tetrachlorodibenzo-*p*-dioxin (TCDD), tetrachlorodibenzofurans (TCDF) or PAHs. If these analytes are found, the filters are re-extracted until the analyte is not detected. The filters were then dried completely under a clean nitrogen (N<sub>2</sub>) stream. Each filter was individually checked for holes, tears, creases or discoloration, and if any were found, was discarded. Acceptable filters were stored in pre-cleaned petri dish, labeled by date of analyses and sealed with Teflon® tape.

**Table 5-5. Method 23 Glassware Cleaning Procedure (Train Components, Sample Containers and Laboratory Glassware)**

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NOTE: USE VITON® GLOVES AND ADEQUATE VENTILATION WHEN RINSING WITH SOLVENTS

1. Soak all glassware in hot soapy water (Alconox®).
2. Tap water rinse to remove soap.
3. Distilled/deionized H<sub>2</sub>O rinse (X3).<sup>a</sup>
4. Bake at 450°F for 2 hours.<sup>b</sup>
5. Acetone rinse (pesticide grade) (X3).
6. Methylene chloride (pesticide grade) (X3).
7. Cap glassware with clean glass plugs or methylene chloride rinsed aluminum foil.
8. Mark cleaned glassware with color-coded identification sticker.
9. Glassware is rinsed immediately before using with acetone and methylene chloride (laboratory proof).

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<sup>a</sup> (X3) = three times.

<sup>b</sup> Step (4) has been added to the cleanup procedure to replace the dichromate soak specified in the reference method. ERG has demonstrated in the past that it sufficiently removes organic artifacts. Step 4 is not used for probe liners and non-glass components of the train that cannot withstand 450°F (i.e., Teflon®-coated filter screen and seals, tweezers, Teflon® squeeze bottles, nylon probe and nozzle brushes).

To prepare the absorbing resin, the XAD-2® resin was cleaned in the following sequential order:

- Rinse with HPLC grade water, discard water;
- Soak in HPLC grade water overnight, discard water;
- Extract in Soxhlet with HPLC grade water for 8 hours, discard water;
- Extract with methanol for 22 hours, discard solvent;
- Extract with methylene chloride for 22 hours, discard solvent;
- Extract with methylene chloride for 22 hours, retain an aliquot of solvent for analysis of CDDs, CDFs and PAHs by GC/MS; and
- Dry resin under a clean N<sub>2</sub> stream.

Once the resin was completely dry, it was checked for the presence of methylene chloride, CDDs, CDFs and PAHs as described in Section 3.1.2.3.1 of the reference method. If any analytes are found, the resin is re-extracted. If methylene chloride is found, the resin is dried until the excess solvent is removed. The absorbent is to be used within four weeks of cleaning.

The cleaned XAD-2® resin was spiked before shipment to the field with five CDD/CDF and one PAH internal standards. Due to the special handling considerations required for the internal standards, the spiking was performed by Triangle Laboratories. For convenience and to minimize contamination, Triangle Laboratories also performed the resin and filter cleanup procedures and loaded the resin into the glass traps.

#### 5.2.2.3 Method 23 Sampling Train Preparation

The remaining preparation included calibration and leak checking of all sampling train equipment, including meterboxes, thermocouples, nozzles, pitot tubes, and umbilicals.

Referenced calibration procedures were followed when available. The results were properly documented in a laboratory notebook or project file and retained.

### **5.2.3 Method 23 Sampling Operations**

#### **5.2.3.1 Preliminary Measurements**

Prior to sampling (data collected during presurvey), preliminary measurements were required to ensure isokinetic sampling. These measurements included determining the traverse point locations, performing a preliminary velocity traverse, cyclonic flow check and moisture determination. These measurements were used to calculate a “K factor.” The K factor was used to determine an isokinetic sampling rate from stack gas flow readings taken during sampling.

Measurements were then made of the duct inside diameter, port nozzle length, and the distances to the nearest upstream and downstream flow disturbances. These measurements were then used to determine sampling point locations by following EPA Reference Method 1 guidelines. The distances were then marked on the sampling probe using an indelible marker.

#### **5.2.3.2 Assembling the Train**

The assembly of the Method 23 sampling train components was completed in the recovery trailer and final train assembly was performed at the stack location. First, the empty, clean impingers were assembled and laid out in the proper order in the recovery trailer. Each ground glass joint was carefully inspected for hairline cracks. The first impinger was a knockout impinger which has a short tip. The purpose of this impinger was to collect condensate which formed in the coil and XAD-2® resin trap. The next two impingers were modified tip impingers which each contained 100 mL of HPLC grade water. The fourth impinger was empty, and the fifth impinger contained 200 to 300 grams of blue indicating silica gel. After the impingers were loaded each impinger was weighed, the initial weight and contents of each

impinger were recorded on a recovery data sheet. The heights of all the impingers were approximately the same to obtain a leak free seal. The open ends of the train were sealed with methylene chloride-rinsed aluminum foil, or clean ground glass caps.

The filter was loaded into the filter holder in the recovery trailer. The filter holder was then capped off and placed with the resin trap and condenser coil (capped) into the impinger bucket. A supply of precleaned foil and socket joints was also placed in the bucket in a clean plastic bag for the convenience of the samplers. Sealing greases were not used thus avoiding contamination of the sample. The train components were transferred to the sampling location and assembled as previously shown in Figure 5-4.

### 5.2.3.3 Sampling Procedures

After the train was assembled, the heaters were turned on for the probe liner and heated filter box and the sorbent module/condensor coil recirculating pump was turned on. When the system reached the appropriate temperatures, the sampling train was ready for pre-test leakchecking. The temperature of the sorbent module resin must not exceed 50°C (120°F) at any time and during testing it must not exceed 20°C (68°F). The filter temperature was maintained at 120 ±14°F (248 ±25°F). The probe temperature was maintained above 100°C (212°F).

The sampling trains were leak checked at the start and finish of sampling. (Method 5/23 protocol only requires post-test leakchecks and recommends pre-test leakchecks.) ERG protocol also incorporates leak checks before and after every port change. An acceptable pre-test leak rate is less than 0.02 acfm (ft<sup>3</sup>/min) at approximately 15 inches of Hg. If during testing, a piece of glassware needed to be emptied or replaced, a leak check was performed before the glassware piece was removed, and after the train was re-assembled.

To leak check the assembled train, the nozzle end was capped off and a vacuum of 15 inches Hg was pulled in the system. When the system was evacuated, the volume of gas flowing through the system was timed for 60 seconds. After the leak rate was determined, the cap was slowly removed from the nozzle end until the vacuum dropped off, and then the pump was turned off. If the leak rate requirement was not met, the train was systematically checked by first capping the train at the filter, at the first impinger, etc., until the leak was located and corrected.

After a successful pre-test leak check had been conducted, all train components were at their specified temperatures and initial data were recorded (DGM reading), the test was initiated. Sampling train data were recorded periodically on standard data forms. A checklist for CDD/CDF sampling is included in Table 5-6. A sampling operation that is unique to CDD/CDF sampling is that the gas temperature entering the resin trap must be below 20°C (68°F). The gas was cooled by a water jacket condenser through which ice water was circulated.

The leak rates and sampling start and stop times were recorded on the sampling task log. Also, any other events that occurred during sampling were recorded on the task log such as sorbent module heat excursions, pitot cleaning, thermocouple malfunctions, heater malfunctions or any other unusual occurrences.

At the conclusion of the test run, the sample pump (or flow) was turned off, the probe was removed from the duct, a final DGM reading was taken, and a post-test leak check was completed. The procedure is identical to the pre-test procedure. However, the vacuum should be at least one inch Hg higher than the highest vacuum attained during sampling. An acceptable leak rate is less than 4% of the average sample rate of 0.02 acfm (whichever is lower).

**Table 5-6. CDD/CDF Sampling Checklist**

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Before test starts:

1. Check impinger set to verify the correct order, orientation and number of impingers. Verify probe markings, and remark if necessary.
  2. Check that you have all the correct pieces of glassware. Have a spare probe liner, probe sheath, meter box and filter ready to go at location.
  3. Check for data sheets and barometric pressure.
  4. Bag sampling equipment for CO<sub>2</sub>/O<sub>2</sub> needs to be ready except when using CEMs for CO<sub>2</sub>/O<sub>2</sub> determinations.
  5. Examine meter box - level it, zero the manometers and confirm that the pump is operational.
  6. Verify the filter is loaded correctly and as tightly as possible; place filter in line with the train and leak check at 15 inches Hg.
  7. Add probe to train.
  8. Check thermocouples - make sure they are reading correctly.
  9. Conduct pitot leak check, recheck manometer zero.
  10. Do final leak check; record leak rate and vacuum on sampling log.
  11. Turn on variacs and check to see that the heat is increasing.
  12. Check that cooling water is on and flowing. Add ice to impinger buckets.
  13. Check isokinetic K-factor - make sure it is correct. (Refer to previous results to confirm assumptions. Two people should calculate this independently to double check it.)
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**Table 5-6. Continued**

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During Test:

1. Notify crew chief of any sampling problems ASAP. Train operator should fill in sampling log and document any abnormalities.
2. Perform simultaneous/concurrent testing with other locations (if applicable). Maintain filter temperature between  $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$ . Keep temperature as steady as possible. Maintain the resin trap and impinger temperatures below  $68^{\circ}\text{F}$ . Maintain probe temperature above  $212^{\circ}\text{F}$ .
3. Leak check between ports and record on data sheet. Leak check if the test is stopped to change silica gel, to decant condensate, or to change filters.
4. Record sampling times, rate, and location for the fixed gas bag sampling ( $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{O}_2$ ), if applicable.
5. Blow back pitot tubes periodically if moisture entrapment is expected.
6. Change filter if vacuum suddenly increases or exceeds 15 inches Hg.
7. Check impinger solutions every 1/2 hour; if the knockout impinger is approaching full, stop test and empty it into a pre-weighed bottle and replace it in the train.
8. Check impinger silica gel every 1/2 hour; if indicator color begins to fade, request a prefilled, preweighed impinger from the recovery trailer.
9. Check the ice in the impinger bucket frequently. If the stack gas temperatures are high, the ice will melt at the bottom rapidly. Maintain condenser coil and silica gel impinger gas temperatures below  $20^{\circ}\text{C}$  ( $68^{\circ}\text{F}$ ).

After test is completed:

1. Record final meter reading.
  2. Do final leak check of sampling train at maximum vacuum during test.
  3. Do final pitot leak check.
-

**Table 5-6. Continued**

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4. Check completeness of data sheet. Verify the impinger bucket identification is recorded on the data sheets. Note any abnormal conditions.
  5. Leak check, check functions (level, zero, etc.) of pitot tubes and inspect for tip damage.
  6. Disassemble train, cap sections, and take each section and all data sheets down to recovery trailer.
  7. Probe recovery (use 950 mL bottles)
    - a) Bring probes into recovery trailer (or other enclosed area).
    - b) Wipe the exterior of the probe to remove any loose material that could contaminate the sample.
    - c) Carefully remove the nozzle/probe liner and cap it off with prerinsed aluminum foil.
    - d) For acetone rinses (all trains)
      - Attach precleaned cyclone flask to probe to catch rinses
      - Wet all sides of probe interior with acetone
      - While holding the probe in an inclined position, put precleaned probe brush down into probe and brush it in and out
      - Rinse the brush, while in the probe, with acetone
      - Do this at least 3 times until all the particulate has been recovered.
      - Recover acetone into a preweighed, prelabeled sample container
    - e) Follow the procedure outlined in (d) using methylene chloride. Recover the solvent into the same acetone recovery bottle.
    - f) Follow the procedure outlined in (d) using toluene. Recover this solvent into a separate preweighed prelabeled sample container.
  8. Cap both ends of nozzle/probe liner for the next day, and store in dry safe place.
  9. Make sure data sheets are completely filled out, legible, and give them to the Crew Chief.
- 
-

#### **5.2.4 CDD/CDF/PAH Sample Recovery**

To facilitate transfer from the sampling location to the recovery trailer, the sampling train was disassembled into the following sections: the probe liner, filter holder, filter to condenser glassware, condenser sorbent module, and the impingers in their bucket. Each of these sections was capped with methylene chloride rinsed aluminum foil or ground glass caps before removal to the recovery trailer. Once in the trailer, field recovery followed the scheme in Figure 5-5. The samples were recovered and stored in cleaned amber glass bottles to prevent light degradation.

The probe and nozzle was first rinsed with approximately 100 mL of acetone and brushed to remove any particulate. This first rinse was followed with a rinse of methylene chloride. Both of these rinses were collected in the same bottle. The same two solvents were used to rinse the cyclone, front/back half filter holder, filter support, connecting glassware and condenser. These rinses were added to the probe rinse bottle. All of the components listed above were again rinsed with toluene, but collected in a separate container.

The contents of impingers 1-4 (H<sub>2</sub>O) were collected in a separate bottle along with their methylene chloride rinses.

The solvents used for train recovery were all pesticide grade. The use of the highest grade reagents for train recovery was essential to prevent the introduction of chemical impurities which interfere with the quantitative analytical determinations.

The train components recovered in the field are listed in Table 5-7. The sorbent module was stored in coolers on ice at all times. The samples were delivered to the analytical laboratory upon return to ERG accompanied by written information designating target analyses.



5-37

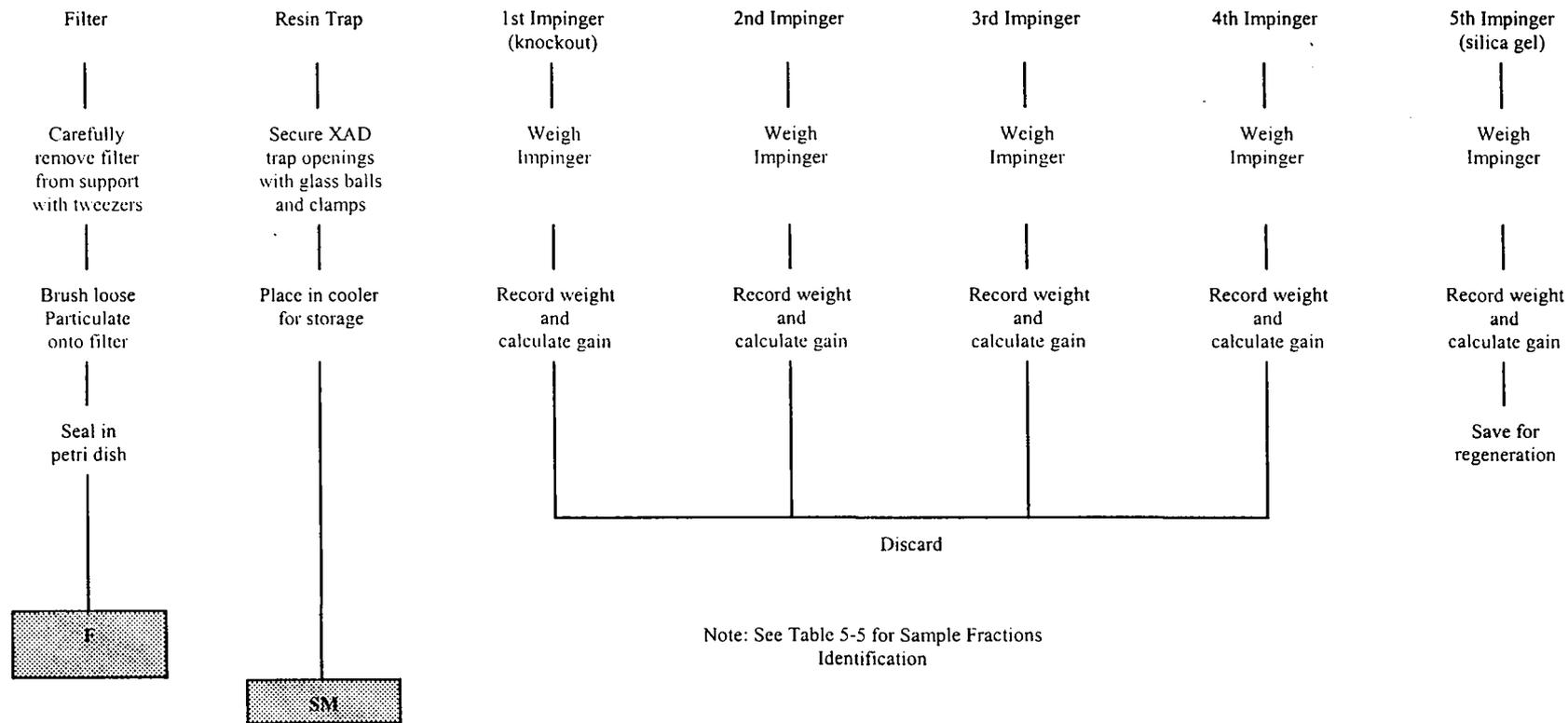


Figure 5-5. Continued

**Table 5-7. Method 23 Sample Fractions Shipped To Analytical Laboratory**

Container/ Component	Code	Fraction
1	F	Filter(s)
2	Pr <sup>a</sup>	Acetone and methylene chloride rinses of nozzle/probe, cyclone, front half/back filter holder, filter support, connecting glassware, condenser
3	PRT CRT	Toluene rinse of nozzle/probe, cyclone, front half/back half filter holder, filter support, connecting line and condenser
4	SM	XAD-2® resin trap (sorbent module)
5	IC	Contents of Impingers 1-4 (H <sub>2</sub> O) plus methylene chloride rinses

<sup>a</sup> Rinses include acetone and methylene chloride recovered into the same sample bottle.

### 5.2.5 CDD/CDF/PAH Analytical Procedures

The analytical procedure used to obtain analyte concentrations from a single flue gas sample is high resolution gas chromatography (HRGC) and high resolution mass spectrometry (HRMS) (resolution from 8000-10000 m/z). The target CDD/CDF congeners are listed in Table 5-8. The PAH analytes are listed in Table 5-9. The analyses were performed by Triangle Laboratories, Inc., by Method 23/8290.

The Method 23 samples were prepared and analyzed according to the scheme in Figure 5-6. The XAD-2® (along with the acetone/methylene chloride rinses) was extracted with methylene chloride and this extract was added to the extract from the extraction of the impinger water. This combined extract was split 1:1, with one half being added to the toluene rinses and toluene extract of the XAD® for D/F analysis, and the remaining being used for PAH analysis. For the D/F analysis, isotopically-labeled surrogate compounds and internal standards and surrogates that were used are described in detail in EPA Method 23.

**Table 5-8. CDD/CDF Congeners To Be Analyzed**

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DIOXINS:

2,3,7,8-tetrachlorodibenzo-*p*-dioxin ( 2,3,7,8-TCDD)  
Total tetrachlorinated dibenzo-*p*-dioxins (TCDD)  
1,2,3,7,8-pentachlorodibenzo-*p*-dioxin (1,2,3,7,8-PeCDD)  
Total pentachlorinated dibenzo-*p*-dioxins (PeCDD)  
1,2,4,5,7,8-hexachlorodibenzo-*p*-dioxin (1,2,3,4,7,8-HxCDD)  
1,2,3,6,7,8-hexachlorodibenzo-*p*-dioxin (1,2,4,5,7,8-HxCDD)  
1,2,3,7,8,9-hexachlorodibenzo-*p*-dioxin (1,2,3,7,8,9-HxCDD)  
Total hexachlorinated dibenzo-*p*-dioxins (HxCDD)  
1,2,3,4,6,7,8-heptachlorodibenzo-*p*-dioxin (1,2,3,4,6,7,8-HpCDD)  
Total heptachlorinated dibenzo-*p*-dioxins (HpCDD)  
Total octachlorinated dibenzo-*p*-dioxins (OCDD)

FURANS:

2,3,7,8-tetrachlorodibenzofurans (2,3,7,8-TCDF)  
Total tetrachlorinated dibenzofurans (TCDF)  
1,2,3,7,8-pentachlorodibenzofuran (1,2,3,7,8-PeCDF)  
2,3,4,7,8-pentachlorodibenzofuran (2,3,4,7,8-PeCDF)  
Total pentachlorinated dibenzofurans (PeCDF)  
1,2,3,4,7,8-hexachlorodibenzofuran (1,2,3,4,7,8-HxCDF)  
1,2,3,6,7,8-hexachlorodibenzofuran (1,2,3,6,7,8-HxCDF)  
2,3,4,6,7,8-hexachlorodibenzofuran (2,3,4,6,7,8-HxCDF)  
1,2,3,7,8,9-hexachlorodibenzofurans (1,2,3,7,8,9-HxCDF)  
Total hexachlorinated dibenzofurans (HxCDF)  
1,2,3,4,6,7,8-heptachlorodibenzofuran (1,2,3,4,6,7,8-HpCDF)  
1,2,3,4,7,8,9-heptachlorodibenzofuran (1,2,3,4,7,8,9-HpCDF)  
Total heptachlorinated dibenzofurans (HpCDF)  
Total octachlorinated dibenzofurans (OCDF)

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**Table 5-9. PAH to be Analyzed**

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Acenaphthene  
Acenaphthylene  
Anthracene  
Benzo(a)anthracene  
Benzo(b)fluoranthrene  
Benzo(k)fluoranthrene  
Benzo(g,h,i)perylene  
Benzo(e)pyrene  
2-Chloronaphthalene  
Chrysene  
Dibenzo(a,b)anthracene  
Fluorene  
Fluorene  
Indeno(1,2,3-cd)pyrene  
2-Methylnaphthalene  
Naphthalene  
Perylene  
Phenanthrene  
Pyrene

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ORGANICS SS SPIKE-I

D5-PHENOL 100 ug  
1,4-DIBROMOBENZENE-D4 100 ug

ORGANICS SS SPIKE-II

D5-NITROBENZENE 100 ug  
2-FLUOROBIPHENYL 100 ug  
1,3,5-TRICHLORO-  
BENZENE-D3 100 ug

ORGANICS SS SPIKE-III

2,4,6-TRIBROMOPHENOL 100 ug

ORGANICS SS SPIKE-IV

ANTHRACENE-D10 100 ug

ORGANICS SS SPIKE-V

PYRENE-D10 100 ug

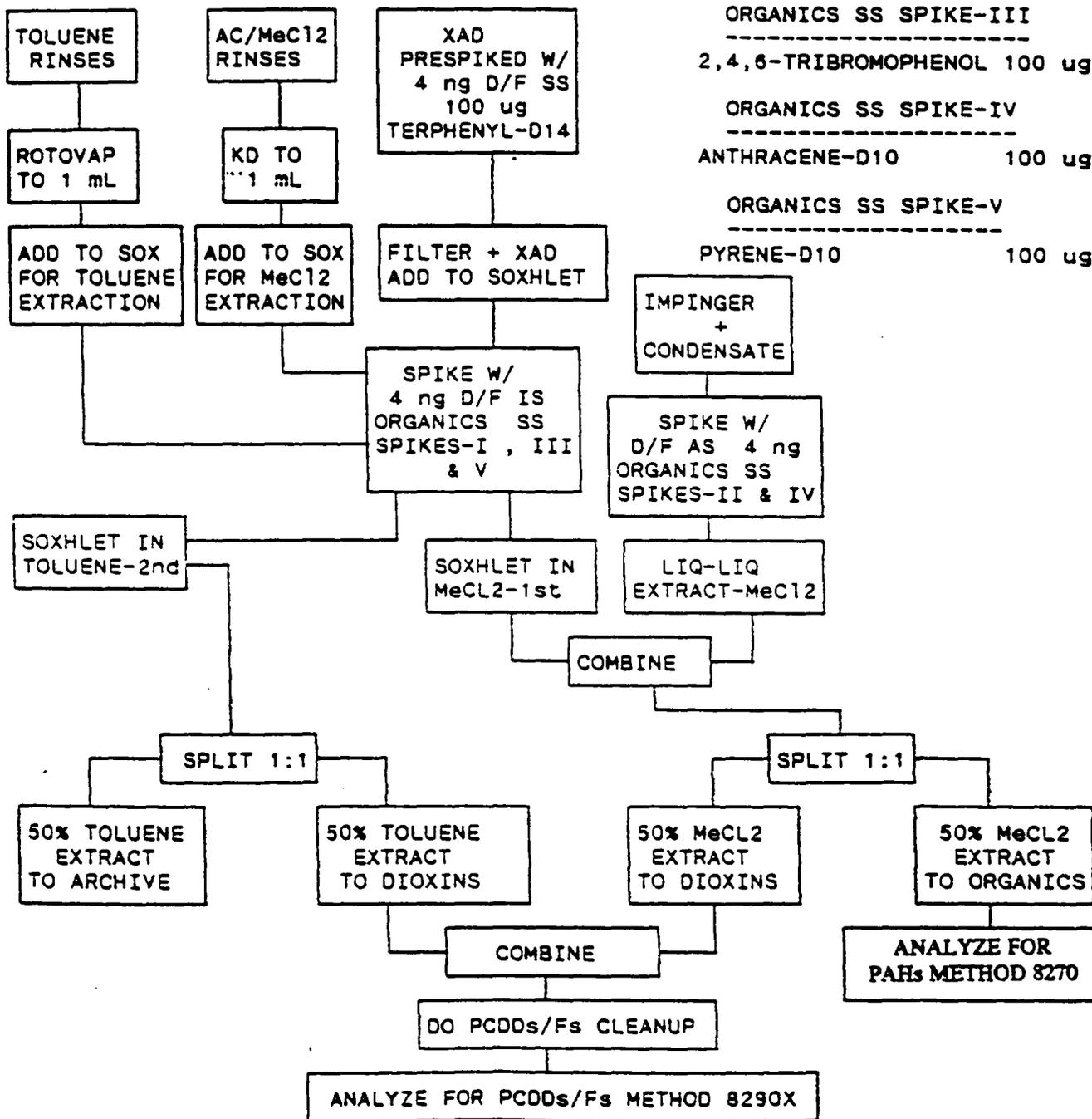


Figure 5-6. Extraction and Analysis Schematic for Method 23 Samples

Data from the mass spectrometer were recorded and stored on a computer file as well as printed on paper. Results such as amount detected, detection limit, retention time, and internal standard and surrogate standard recoveries were calculated by computer. The chromatograms were retained by the analytical laboratory with copies included in the analytical report delivered to ERG.

#### 5.2.5.1 Preparation of Samples for Extraction

Upon receiving the sample shipment, the samples were checked against the Chain-of-Custody forms and then assigned an analytical laboratory sample number. Each sample component was reweighed to determine if leakage occurred during travel. Color, appearance, and other particulars of the samples were noted. Samples were extracted within 21 days of collection and processed through cleanup procedures before concentration and analysis.

#### 5.2.5.2 Calibration of GC/MS System

A five-point calibration of the GC/MS system was performed to demonstrate instrument linearity over the concentration range of interest. Relative response factors were calculated for each congener or compound of interest. The response factors were verified on a daily basis using a continuing calibration standard consisting of a mid-level isomer standard. The instrument performance was acceptable only if the measured response factors for the labeled and unlabeled compounds and the ion-abundance ratios were within the allowable limits specified in the method.

#### 5.2.6 *CDD/CDF Analytical Quality Control*

All quality control procedures specified in the test method were followed. Blanks were used to determine analytical contamination, calibration standards were used for instrument calibration and linearity checks, internal standards were used to determine isomer recoveries and

adjust response factors for matrix effects, surrogate standards were used to measure the collection efficiency of the sampling methodology and an alternate standard was used as a column efficiency check.

### 5.2.6.1 CDD/CDF Quality Control Blanks

Four different types of sample blanks were collected for D/F analysis. The type of blanks that are required are shown in Table 5-10.

**Table 5-10. Method 23 Blanks Collected**

<b>Blank</b>	<b>Collection</b>	<b>Analysis</b>
Field Blanks	One run collected and analyzed	Analyze with flue gas samples
Glassware Proof Blank	Each train to be used (2) will be loaded and quantitatively recovered prior to sampling	Archive for potential analysis
Method Blank	At least one for each analytical batch	Analyze with each analytical batch of flue gas samples
Reagent Blanks	One 1000 mL sample for each reagent and lot	Archive for potential analysis

Reagent blanks of 1000 mL of each reagent used at the test site were saved for potential analysis. Each reagent blank was of the same lot as was used during the sampling program. Each lot number and reagent grade was recorded on the field blank label and in the laboratory notebook (acetone, methylene chloride, toluene, HPLC water, filter, XAD-2®).

A glassware blank (proof blank) was recovered from each set of sample train glassware that was used to collect the organic samples. The precleaned glassware, which consists of a probe liner, filter holder, condensor coil, and impinger set, was loaded as if for sampling and then quantitatively recovered exactly as the samples were. Analysis of the generated fractions were

used to check the effectiveness of the glassware cleaning procedure only if sample analysis indicates a potential contamination problem.

A field blank was collected from a set of D/F glassware that had been used to collect at least one sample and had been recovered. The train was re-loaded, leak checked and left at a sampling location during a test run. The train was then recovered. The purpose of the field blank was to measure the level of contamination that occurs from handling, loading, recovering, and transporting the sampling train. The field blanks were analyzed with the flue gas samples. If they are unsatisfactory in terms of contamination, reagent blanks may be analyzed to determine the specific source of contamination.

In addition to the three types of blanks that are required for the sampling program, the analytical laboratory analyzed a method blank with each set of flue gas samples. This method blank consisted of preparing and analyzing an aliquot of toluene by the exact procedure used for the samples analysis. The purpose of this method blank was to verify that there was no laboratory contamination of the field samples.

#### 5.2.6.2 Quality Control Standards and Duplicates

Recoveries of the internal standards must be between 40 to 130% for the tetra-through hexachlorinated compounds and in the range of 25 to 130% for the hepta-and octachlorinated homologues. If these requirements are not met, the data will be acceptable if the signal to noise ratio is greater than or equal to ten. If these requirements are met, the results for the native (sampled) species are adjusted according to the internal standard recoveries.

Surrogate standard recoveries must be between 70 to 130%. If the recoveries of all standards are less than 70%, the project director will be notified immediately to determine if the surrogate results will be used to adjust the results of the native species.

### **5.2.7 Analytes and Detection Limits for Method 23**

The target analytes are the tetra- through octachlorinated dibenzodioxins and chlorinated dibenzofurans. The detection limit of the individual compounds is dependent on the detection limit of the analytical method, the volume of the final extract and the total volume of gaseous sample collected in the sampling trains. Following the protocol of Method 23, the fractions to be collected for analysis from each train are:

- Fraction 1--Filter;
- Fraction 2--XAD-2® sorbent module;
- Fraction 3--Acetone and methylene chloride rinses of all train components prior to sorbent module and;
- Fraction 4--Toluene rinses of all train components prior to the sorbent module.
- Fraction 5--Impinger contents 1-4 plus methylene chloride rinses

Following the sample preparation protocol outlined in Method 23, a single combined sample was presented for analysis for D/F by high resolution gas chromatography/high resolution mass spectrometry. (The individual samples were no longer available for analysis). The final volume of this sample was 200  $\mu\text{L}$  of which a 2  $\mu\text{L}$  aliquot was injected into the instrument. Using an instrument detection limit of 50 pg for tetra-, 250 pg for penta- through hepta-, and 500 pg for octa-, the total minimum detectable amounts were calculated and are given in Table 5-11. Using a four hour sampling time as selected by the EPA Work Assignment Manager at an assumed sampling rate of 0.75 cfm, the MDLs shown in Table 5-12 were possible. The sampling flow rate at the outlet location was dictated by the flow rate of the stack gas since isokinetic sampling was performed.

**Table 5-11. Analytical Detection Limits For Dioxins/Furans**

Analyte	Total Detectable Amount, ng
Tetra CDDs	5
Penta CDDs	25
Hexa CDDs	25
Hepta CDDs	25
Octa CDDs	50
Tetra CDFs	5
Penta CDFs	25
Hexa CDFs	25
Hepta CDFs	25
Octa CDFs	50

NOTE: D/F analysis by High Resolution Mass Spectrometry assumes a 2  $\mu\text{L}$  injection of a 200  $\mu\text{L}$  sample extract.

**Table 5-12. CDD/CDF Method Detection Limits**

Sampling Time, Hours	4
Sampling Rate, cfm	0.75
Sample Volume, m <sup>3</sup>	5.1
	MDL,ng/m <sup>3</sup>
Tetra CDDs	0.98
Penta CDDs	4.9
Hexa CDDs	4.9
Hepta CDDs	4.9
Octa CDDs	9.8
Tetra CDFs	0.98
Penta CDFs	4.9
Hexa CDFs	4.9
Hepta CDFs	4.9
Octa CDFs	9.8

### 5.3 Analysis of Method 23 Samples for PAHs

The Method 23 sample preparation scheme shown in Figure 5-6 includes the splitting of prepared sample extracts for both CDD/CDF and PAH analyses. Split extracts were analyzed for the PAH compounds shown in Table 5-9 using gas chromatography coupled with high resolution mass spectrometry. However, due to high levels of some PAHs, the extracts were re-analyzed using low resolution mass spectrometry. Table 5-13 lists the analytical detection limits for each of the PAHs to be determined.

**Table 5-13. Analytical Detection Limits For PAHs**

Analyte	Total Detectable Amount, $\mu\text{g}$
Acenaphthene	20
Acenaphthylene	10
Anthracene	10
Benzo(a)anthraene	10
Benzo(b)fluoranthene	10
Benzo(k)fluorethene	10
Benzo(g,h,i)perylene	10
Benzo(a)pyrene	10
Benzo(e)pyrene	10
2-Chloronaphthalene	10
Chrysene	10
Dibenzo(a,h)anthracene	10
Fluoranthene	20
Fluorene	35
Indeno(1,2,3-cd)pyrene	10
2-Methylnaphthalene	150
Naphthalene	900
Perylene	10
Phenanthrene	100
Pyrene	15

Using a four hour sampling time as selected by the EPA Work Assignment Manager, at an assumed sampling rate of 0.75 cfm, the method detection limits shown in Table 5-14 were possible.

**Table 5-14. PAH Method Detection Limits**

Sampling Time, Hours	4
Sampling Rate, cfm	0.75
Sample Volume, m <sup>3</sup>	5.1
	MDL, $\mu\text{g}/\text{m}^3$
Acenaphthene	8
Acenaphthylene	4
Anthracene	4
Benzo(a)anthracene	4
Benzo(b)fluoranthene	4
Benzo(k)fluoranthene	4
Benzo(g,h,i)perylene	4
Benzo(a)pyrene	4
Benzo(e)pyrene	4
2-Chloronaphthalene	4
Chrysene	4
Dibenzo(a,h)anthracene	4
Fluoranthene	8
Fluorene	14
Indeno(1,2,3-cd)pyrene	4
2-Methylnaphthalene	59
Naphthalene	350
Perylene	4
Phenanthrene	40
Pyrene	6

## **5.4 EPA Methods 1-4**

### **5.4.1 *Traverse Point Location By EPA Method 1***

The number and location of sampling traverse points necessary for isokinetic and flow sampling were dictated by EPA Method 1 protocol. These parameters were based upon how much duct distance separates the sampling ports from the closest downstream and upstream flow disturbances. The minimum number of traverse points for a circular duct with an I.D. of 12 feet is 12.

### **5.4.2 *Volumetric Flow Rate Determination by EPA Method 2***

Volumetric flow rate was measured according to EPA Method 2. A type K thermocouple and S-type pitot tube were used to measure flue gas temperature and velocity, respectively.

#### **5.4.2.1 Sampling and Equipment Preparation**

For EPA Method 2, the pitot tubes were calibrated before use following the directions in the method. Also, the pitots were leak checked before and after each run.

#### **5.4.2.2 Sampling Operations**

The parameters that were measured include the pressure drop across the pitots, stack temperature, stack static and ambient pressure. These parameters were measured at each traverse point, as applicable. A computer program was used to calculate the average velocity during the sampling period.

#### **5.4.3 *O<sub>2</sub> and CO<sub>2</sub> Concentrations by EPA Method 3***

The O<sub>2</sub> and CO<sub>2</sub> concentrations were determined by Fyrite following EPA Method 3. Flue gas was extracted from the duct for analysis. The Method 3 analysis for O<sub>2</sub> and CO<sub>2</sub> were performed approximately every 30 minutes as a grab sample at the outlet and at the inlet.

#### **5.4.4 *Average Moisture Determination by EPA Method 4***

The average flue gas moisture content was determined according to EPA Method 4. Before sampling, the initial weight of the impingers was recorded. When sampling was completed, the final weights of the impingers were recorded, and the weight gain was calculated. The weight gain and the volume of gas sampled were used to calculate the average moisture content (%) of the flue gas. The calculations were performed by computer. Method 4 was incorporated in the technique used for the Method 29 manual sampling method that was used during the test.



## **6.0 QUALITY ASSURANCE/QUALITY CONTROL**

Specific Quality Assurance/Quality Control (QA/QC) procedures were strictly followed during this test program to ensure the production of useful and valid data throughout the course of the project. A detailed presentation of QC procedures for all sampling and analysis activities can be found in the Site Specific Test Plan and Quality Assurance Project Plan for this project. This section reports the results of all QC analyses so that the degree of data quality can be ascertained.

In summary, a high degree of data quality was maintained throughout the project. All sampling train leak checks met the QC criteria as specified in the methods. Isokinetic sampling rates were kept within the 10% of 100% for all test runs. Good spike recoveries and close agreement between duplicate analyses were shown for the sample analyses.

### **6.1 Sampling QC Results**

The following sections discuss the QC results of the specific sampling methods employed during this project.

#### **6.1.1 D/F/PAH Sampling QC**

Table 6-1 lists the pre- and post-test and port change leak check results. The acceptance criteria are that all post-test leak checks must be less than 0.02 cfm or 4 percent of the average sampling rate (whichever is less). All D/F/PAH leak checks met this criterion.

Table 6-2 presents the isokinetic sampling rates for the D/F/PAH sampling runs. The acceptance criterion is that the average sampling rate must be within 10% of 100% isokinetic. All sampling runs met this criterion.

**Table 6-1. Summary of Leak Checks Performed, Per Port, Dioxin Testing, Outlet**

<b>Date</b>	<b>Run #/Port</b>	<b>Initial leak Check</b>	<b>Leak Check</b>	<b>Final Leak Check</b>
6/25/97	1/C	0.015 @ 17"	0.011 @ 10"	
	1/D		0.016 @ 12"	
	1/A		0.017 @ 10"	
	1/B		0.018 @ 15"	
6/26/97	2/B	0.009 @ 17"	0.007 @ 10"	
	2/A		0.008 @ 7"	
	2/D		0.009 @ 9"	
	2/C		0.011 @ 9"	
6/27/97	3/C	0.010 @ 10"	0.003 @ 12"	
	3/D		0.009 @ 12"	
	3/A		OK	
	3/B		0.011 @ 10"	

**Table 6-2. Summary of Isokinetic Percentages**

<b>Date</b>	<b>Run #</b>	<b>Percent Isokinetic</b>
<b>Multi-Metals -- Inlet</b>		
6/25/97	1	102.66
6/26/97	2	108.38
6/27/97	3	99.42
<b>Multi-Metals -- Outlet</b>		
6/25/97	1	103.74
6/26/97	2	99.66
6/27/97	3	103.25
<b>Dioxin -- Outlet</b>		
6/25/97	1	104.38
6/26/97	2	103.67
6/27/97	3	102.29

All dry gas meters are fully calibrated every six months against an EPA approved intermediate standard. The full calibration factor is used to correct the actual metered sample volume to the true sample volume. To verify the full calibration, a post-test calibration is performed. The full and post-test calibrations coefficients must be within 5% to meet ERG's internal QA/QC acceptance criterion. As shown in Table 6-3, the meter box used for the D/F/PAH testing met this criterion.

Field blanks are collected to verify the absence of any sample contamination. A D/F/PAH train was assembled as if for sampling, leak checked at the sampling location, left at the sampling location for the duration of a test run and then recovered. Table 6-4 presents the analytical results for the field blank as well as the laboratory method blank. The only D/F compounds detected in the field blank were 1,2,3,4,6,7,8,9-OCDD, 2,3,7,8-TCDF and 1,2,3,4,7,8-HxCDF, but at much lower amounts than in any of the test runs. Any PAHs detected in the field blank were at levels less than 0.5% of any detected in the test runs. Because the amount of contamination was so low, no blank corrections were made on the emissions results.

### **6.1.2 Metals/PM Sampling QC**

Tables 6-5 and 6-6 list the pre- and post-test and port change leak check results for the outlet and inlet sampling trains respectively. The acceptance criteria of less than 0.02 cfm or 4% of the average sampling rate (whichever is less) were met by all sampling trains.

Table 6-2 presents the isokinetic sampling rates for the metals/PM sampling runs. The sampling rate acceptance criterion of being within 10% of 100% isokinetic was met for all sampling runs at both the inlet and outlet.

As shown in Table 6-3, the calibration coefficients of the meter boxes used for the metals/PM testing were within 5% of their full calibration coefficient, thus meeting the acceptance criterion.

**Table 6-3. Dry Gas Meter Post Calibration Results**

<b>Sampling Train</b>	<b>Meter Box Number</b>	<b>Full Calibration Factor</b>	<b>Post-Test Calibration Factor</b>	<b>Post-Test* Deviation %</b>
D/F/PAH, Outlet	39	0.996	0.970	-2.61
Metals/PM, Outlet	38	0.984	0.971	-1.32
Metals/PM, Inlet	40	0.984	0.974	-1.02

\*

$$\frac{\text{Post-Test} - \text{Full}}{\text{Full}} \times 100$$

**Table 6-4. Dioxin/Furan Field Blank Analysis Results**

<b>Congener</b>	<b>Field Blank ng Detected</b>	<b>Lab Method Blank ng detected</b>
2,3,7,8 -TCDD	<0.03*	<0.01
1,2,3,7,8-PeCDD	<0.03	<0.02
1,2,3,4,7,8-HxCDD*	<0.02	<0.02
1,2,3,6,7,8-HxCDD	<0.02	<0.01
1,2,3,7,8,9-HxCDD	<0.04	< 0.02
1,2,3,4,6,7,8--HpCDD	<0.04	0.02
1,2,3,4,6,7,8,9-OCDD*	0.10	<0.04*
2,3,7,8-TCDF**	0.02	<0.007
1,2,3,7,8-PeCDF	<0.02	<0.01
2,3,4,7,8-PeCDF	<0.02	<0.01
1,2,3,4,7,8-HxCDF	0.02	<0.02*
1,2,3,6,7,8-HxCDF	<0.01	<0.01
2,3,4,6,7,8-HxCDF	<0.02	<0.02
1,2,3,7,8,9-HxCDF	<0.02	<0.02
1,2,3,4,6,7,8-HpCDF	<0.03	<0.04*
1,2,3,4,7,8,9-HpCDF	<0.03*	<0.03
1,2,3,4,6,7,8,9-OCDF	<0.06*	0.08

\* Maximum value, may include interference from a diphenyl ether

\*\*Determined from DB-225 GC column

**Table 6-5. Summary of Leak Checks Performed, Per Port, Metals Testing, Outlet**

<b>Date</b>	<b>Run #/Port</b>	<b>Initial leak Check</b>	<b>Leak Check</b>	<b>Final Leak Check</b>
6/25/97	1/D	0.015 @ 15"	*0.014 @ 10"	0.007 @ 10"
	1/A	0.015 @ 15"	0.009 @ 10"	
	1/B		0.006 @ 8"	
	1/C		0.007 @ 10"	
6/26/97	2/C	0.013 @ 12"	0.011 @ 9"	0.007 @ 7"
	2/B	0.013 @ 12"	0.010 @ 7"	
	2/A		0.008 @ 7"	
	2/D		0.007 @ 7"	
6/27/97	3/D	0.009 @ 10"	0.004 @ 7"	0.004 @ 8"
	3/A	0.009 @ 10"	0.006 @ 9"	
	3/B		OK	
	3/C		0.004 @ 8"	

\*Volume Correction .04

**Table 6-6. Summary of Leak Checks Performed, Per Port, Metals Testing, Inlet**

<b>Date</b>	<b>Run #/Port</b>	<b>Initial leak Check</b>	<b>Leak Check</b>	<b>Final Leak Check</b>
6/25/97	1/A	0.019 @ 15"	*0.03 @ 1"	0.001 @ 5"
	1/B	0.019 @ 15"	OK	
	1/C		OK	
	1/D		0.001 @ 5"	
6/26/97	2/A	0.01 @ 10"	0.01 @ 3"	0.01 @ 5"
	2/B	0.01 @ 10"	0.01 @ 3"	
	2/C		0.01 @ 3"	
	2/D		0.01 @ 5"	
6/27/97	3/A	0.001 @ 10"	0.01 @ 5"	0.001 @ 5"
	3/B	0.001 @ 10"	0.001 @ 3"	
	3/C		0.001 @ 3"	
	3/D		0.001 @ 5"	

\*Volume Correction .03

Table 6-7 presents the results from the two Method 29 (metals) field blanks, the reagent blank and the average of three runs at both the outlet and inlet. Chromium, manganese, nickel, lead, antimony and selenium were detected in both field blanks and in the reagent blank. The amount of chromium detected in each of the three blanks was the same with the contribution coming almost entirely from the filter. This value represents approximately 10% of that observed in the inlet samples and approximately 50% of that observed in the outlet samples. The amount of manganese detected varied in the three blanks and ranged between 7  $\mu\text{g}$  in the reagent blank to 129  $\mu\text{g}$  in the inlet field blank, twice the amount detected in the outlet samples. These levels could be due to field contamination, but because proportionately elevated levels for the other metals were not detected in the blanks, and Run 3, Outlet had obvious laboratory contamination (see Section 2.3.2), laboratory contamination is also suspected in this case. The same scenario as discussed for manganese also applies to the lead results; however, any contamination, either field or laboratory related, is insignificant ( $<0.2\%$ ) when compared to the amount detected in either the inlet or the outlet samples. The amount of nickel detected in the reagent blank and the inlet field blank was the same (nominal 25  $\mu\text{g}$ ) while the outlet field blank contained approximately 50  $\mu\text{g}$ . Again, the outlet field blank nickel result is probably due to specific laboratory contamination as other metals do not reflect this amount as general field contamination. Using the inlet field blank and reagent blank as being representative of background levels of nickel, the blank contribution to the levels detected in the samples would be approximately 25% of the total for both the inlet and outlet samples. Antimony was present in all three blanks at approximately the same level in each. This amount is approximately the same as that detected in the outlet samples and 50% of that detected in the inlet samples. The same is true for the amount of selenium detected in the three blanks, but the value represents only 16% of that detected in the outlet samples and 11% of that detected in the inlet samples. The analysis results presented in Section 2.2 of this report have not been blank corrected.

**Table 6-7. Metals QC Results: ( $\mu\text{g}$  detected)**

Metal	Outlet Field Blk	Inlet Field Blk	Train Reagent Blk	Average	
				Outlet Runs	Inlet Runs
Hg	<3.03	<3.26	<2.66	6.41	4.06
As	<1.08	<1.00	<1.00.	4.65	18.2
Be	<0.200	<0.200	<0.200	<0.218	<0.224
Cd	<0.200	<0.200	<0.200	72.6	84.3
Co	<0.200	<0.200	<0.200	<0.218	1.24
Cr	12.8	11.9	11.7	22.0	98.7
Mn	29.7	129	6.85	73.2*	1700
Ni	49.5	25.2	23.7	91.9	98.5
Pb	31.1	22.2	2.68	15700	19000
Sb	6.60	6.4	7.35	6.92	11.0
Se	7.10	6.20	6.35	36.8	54.7

\*Average of two runs, apparent lab contamination in one test run

## 6.2 Analytical QC Results

The following section reports QA parameters for the D/F/PAH and Metals/PM analytical results.

### 6.2.1 D/F/PAH Analytical Quality Control

**D/F**—One sample was generated for D/F analysis for each stack gas sample collected and was subjected to both a full screen and confirmation analysis. The full screen analyses were conducted using a DB-5 GC column which allows the separation of each class of chlorinated (i.e., tetra, penta, etc.) and fully resolves 2,3,7,8-TCDD from the other TCDD isomers. The confirmation analysis, performed on a DB-225 GC column, is needed to fully resolve the 2,3,7,8-TCDF from the other TCDF isomers.

A component of the D/F QC program is adding isotopically labeled standards to each sample during various stages of analysis to determine recovery efficiencies and to aid in the quantitation of native D/F species. Four different types of standards are added:

- Surrogate standards are usually spiked on the XAD-2® absorbent prior to sampling. Recovery of these compounds allows for the evaluation of overall sample collection efficiency and analytical matrix effects.
- Internal standards are spiked after sampling but prior to extraction.
- Alternate standards are also spiked at this stage.
- Recovery percentages of internal standards are used in quantifying the D/F native to the stack gas being sampled. Recovery of alternate standards for extraction/fractionation efficiencies to be determined.
- Recovery standards are added after fractionation, just prior to analysis by HRGC/HRMS.

The recovery of each of the spiked isotopically labeled compounds was within the acceptance criteria set forth in Method 23, except for the surrogate standard  $^{13}\text{C}_{12}$ -1,2,3,4,7,8-HxCDD in Run 1 and Run 2 at the venturi outlet. The percent recoveries were 69.3 and 68.8, respectively, and were just outside of the lower limit of 70%. This low recovery will have no effect on the reported results.

**PAH**—The sample extracts were originally analyzed by HRGC/HRMS, but due to the high level of many of the PAHs found in the samples, the instrument detector became saturated, resulting in data that were not reliable. Therefore, the sample extracts were reanalyzed on a low resolution mass spectrometer (LRMS) following the protocol given in EPA Method 8270A. Unfortunately, the isotopically labeled spiking compounds associated with the PAH analysis that were originally spiked at a level commensurate with HRMS were below the detection limit of the LRMS. It is assumed, however, that the acceptable extraction efficiencies demonstrated for the D/F related spiking standards is indicative of similar acceptable extraction efficiencies for the PAH target compounds. All internal standard areas were within Method 8270A quality control criteria.

### **6.2.2 *Metals Analytical Quality Control***

**ICAP Metals**—The analytical methods used for the stack gas samples are discussed in Section 5 of this report. The following paragraphs discuss the metals QC results.

Serial dilutions were performed on the Outlet Run 1 front half and back samples for the ICAP metals. A serial dilution is performed to determine if there is any interference specific to an analyte in the native sample matrix. The relative percent difference (RPD) between the analysis of the undiluted and the serially diluted sample is determined. Only those analytes with detectable amounts above 10 times the reportable detection limit (RDL) after dilution are reported. Cadmium, manganese, nickel, lead and selenium in the front half sample were within

the  $\pm 10\%$  RPD criterion. Chromium had a RPD of 12.1% which does not indicate any significant interferent. Selenium in the back half sample had an RPD of 8.70%.

Duplicate ICAP analysis was performed on Outlet Run 2 front half and back half samples. Only those analytes with detectable amounts above 10 times the reportable detection limit are reported. The RPD between the two analyses must be  $\pm 20\%$  to be acceptable. All of the metals detected above 10 times the RDL demonstrated RPDs less than 10%.

Post digestion matrix spikes were performed on the Outlet Run 1 front half and back samples for the ICAP metals. Each of the target metals is spiked at a known level into an aliquot of the sample. A percent recovery between 75 and 125 is acceptable and indicates the lack of interference from the native sample matrix. The percent recovery for all the metals except cadmium, manganese, nickel and lead in the front half sample were within 75-125% range. The level of spike for these metals was insignificant compared to the native amount and could not be quantitated. The % recovery for all of the metals in the back half sample were within the acceptance criterion.

No ICAP metals were detected in the laboratory method blank above the instrument detection limit and the recoveries of each of the metals in the laboratory control spike were within the acceptance criterion of 80-120%.

**CVAA**—Every sample was analyzed in duplicate for the presence of mercury. All duplicate analyses were within the acceptance criterion of  $\pm 20\%$ . Matrix spikes and matrix spike duplicates were performed on the back-half (nitric acid/peroxide) impinger contents of all test runs, both inlet and outlet. Percent recoveries for all sample spikes were within the acceptance criterion of 75-125. All laboratory control spikes and laboratory control spike duplicates were also within this acceptance criterion.

### **6.2.3 *PM Analytical Quality Assurance***

All filters and acetone probe rinse residues were weighed to a constant weight following the procedures given in EPA Method 5. The acetone probe rinse residues were blank corrected using a known volume of acetone reagent. The five place analytical balance calibration was verified prior to use by weighing a series of Class S weights which covered the range of weights encountered with the samples.

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(Please read Instructions on reverse before completing)

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16. ABSTRACT    Integrated iron and steel manufacturing is among the categories of major sources for which national emission standards for hazardous air pollutants (NESHAPS) are to be issued by November 2000 pursuant to Section 112 of the Clean Air Act. The integrated iron and steel manufacturing category includes mills that produce steel from iron ore. Key processes and unit operations include sinter production, iron production, steel making, continuous casting, and the preparation of semi-finished and finished products. Source tests are required to quantify and hazardous air pollutant (HAP) emissions, and the performance of a sintering plant equipped with characterize the particulate matter (PM), a venturi scrubber.				
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